METALLURGIA

THE BRITISH JOURNAL OF METALS

Vol. 54 No. 322 AUGUST, 1956 Monthly: TWO SHILLINGS

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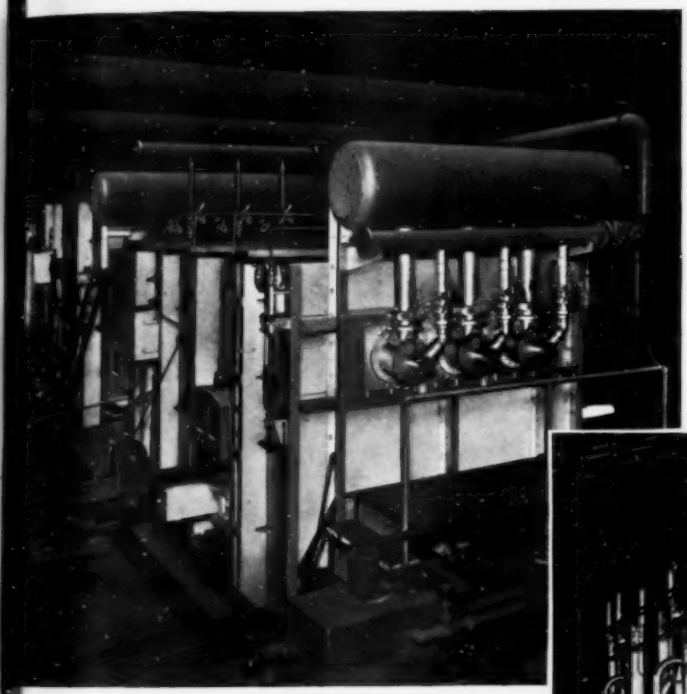

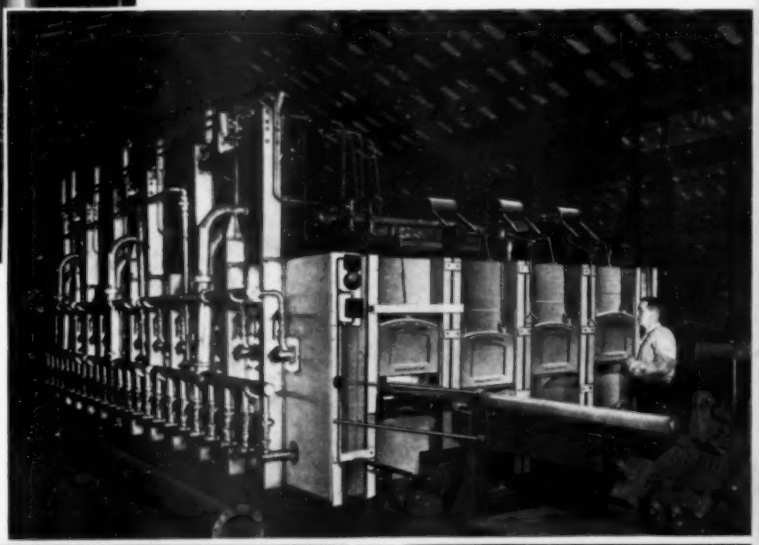

The illustrations show furnaces supplied to the new Tube Mill at Babcock & Wilcox Ltd., at Dumbarton. They include a gas fired Billet Heating Furnace, 6' x 70' (upper illustration). A Roll Down Tube Reheating Furnace, 35' x 10' 9" (centre ...ration). A continuous ...oller Hearth Furnace, ...0' (lower illustration)

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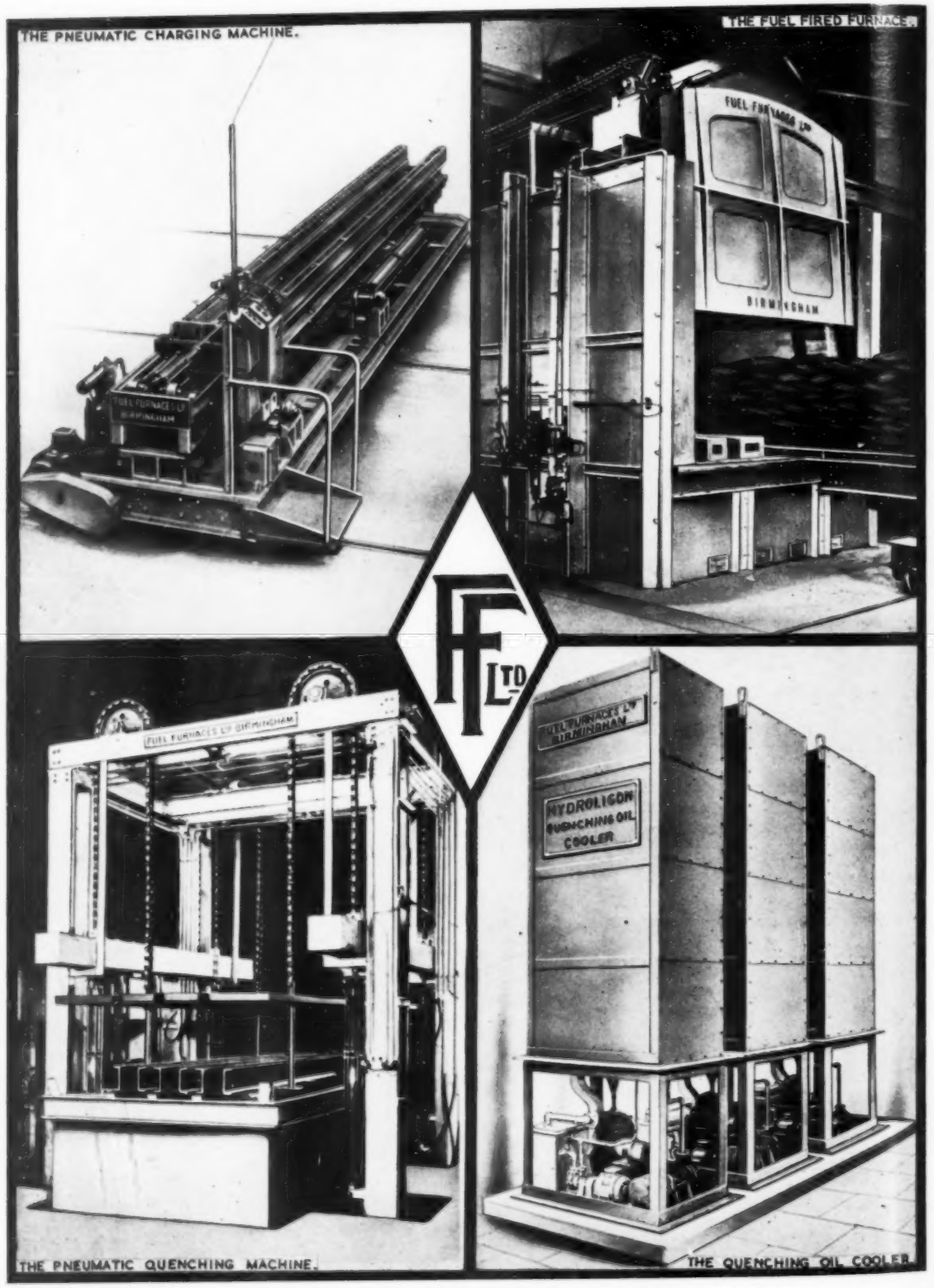
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INCORPORATING THE METALLURGICAL ENGINEER

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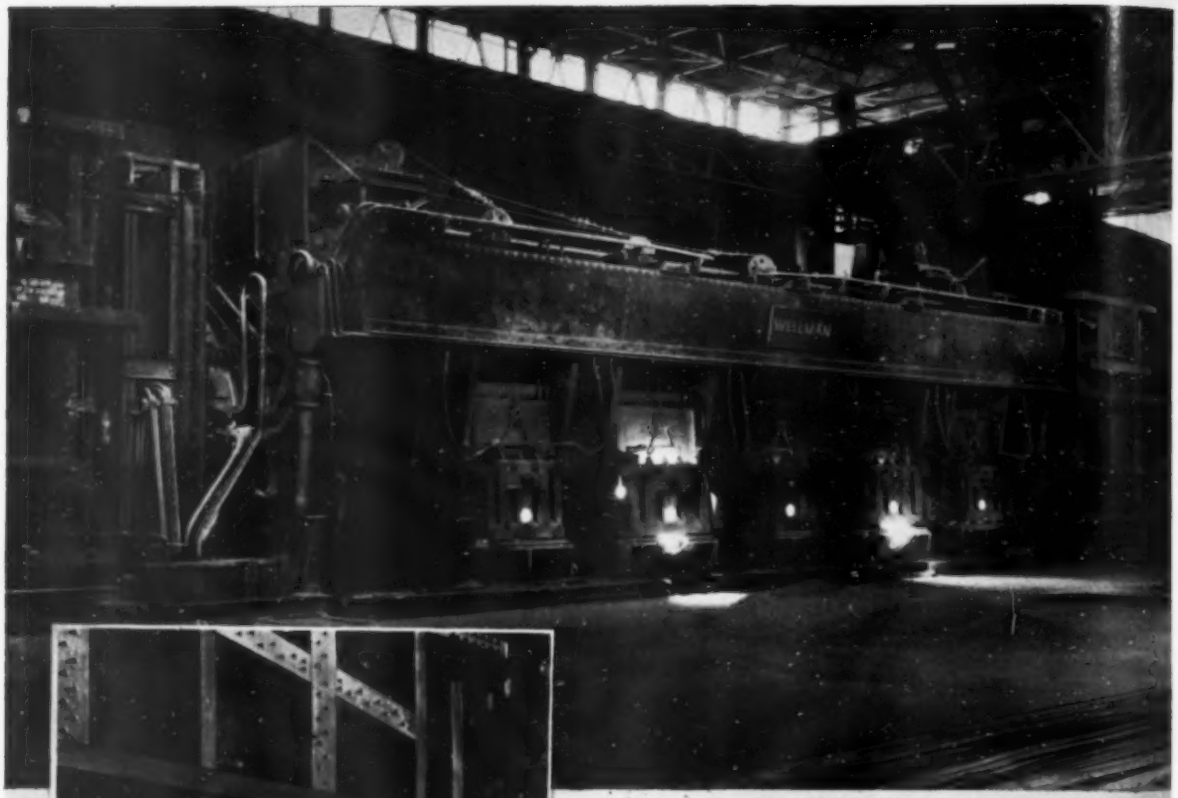

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THE BRITISH JOURNAL OF METALS

INCORPORATING THE "METALLURGICAL ENGINEER"

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German Competition

OVER the last two years, West German imports have expanded by over 40% in volume, and her share in world trade has risen from 13·3% to 15·6%. This rapid expansion cannot be explained—as could to some extent the expansion which occurred before 1953—by the low level from which she started. The increase in Germany's export trade since 1950 has been at the expense of practically all exporting countries* except Japan and Canada, but the United Kingdom has lost much more ground than her other competitors. This was particularly true from 1950 to 1951, when the U.K. share fell from 25·5% to 22·0%, but the fall has continued since 1951, although much more gradually, and the figure for 1955 was 19·8%.

That Germany is becoming an increasingly important competitor of the United Kingdom for the export markets of the world is made abundantly clear by the statistics presented in a recent article in *Board of Trade Journal*†, which goes on to attempt an appraisal of the factors affecting the ability of a country to compete successfully in the export field. This article is based on a report on the impact of German competition, with particular reference to the engineering industries, as it appeared at the beginning of 1956. The Engineering Advisory Council, representative of both sides of the industry, considers the report to be a fair and important statement of the facts, and has recommended that it should be discussed as widely as possible by managements, workers and such bodies as local Productivity Committees.

Perhaps the most obvious factors influencing the ability of a country to compete in world markets are labour costs, raw material costs and fuel costs. The main raw materials for the engineering industries are steel and non-ferrous metals. Steel prices are lower in the United Kingdom than in Germany—in some cases by as much as 20%. Lead and zinc prices are probably about the same in the two countries, and most other non-ferrous metals appear to be rather cheaper in the United Kingdom, although it is not easy to find a common basis on which to compare prices. Coal and electricity appear to be rather cheaper in the United Kingdom, but hard coke prices are a little dearer and fuel oil prices are about the same. There appears to have been no significant change in relative costs of raw materials over the last two years, and the broad conclusion is that over the whole field of raw material and fuel costs some small advantage lies with the United Kingdom.

The evidence points to the labour costs per unit of output in the metal using industries having remained fairly steady since 1950 in Germany, whereas in the United Kingdom they have risen by as much as a quarter. It is impossible to determine, however, how far the resultant cost reduction has been passed on in lower export prices and how far it has been retained as higher profits. Apart from direct wages, labour costs are influenced by paid holidays, social security payments, and productivity, and although there is no reliable evidence as to comparative productivity in the two countries, there is little doubt that the increase in productivity in Germany has been very rapid in the past five years, and that it has been responsible for the favourable trend—from the German point of view—of labour costs over that period, as wages have risen at roughly the same rate in the two countries. Quite apart from any question of effort by the individual worker, the rise in productivity must owe a great deal to the rising rate of expenditure on industrial equipment.

There are, of course, other factors affecting a country's exporting ability, such as the possible effects of taxation ; the availability of capital and credit assurance ; and the pull of the home market. The levels of company taxation in the two countries are very similar, and although income tax rates are rather higher in Germany than they are in the United Kingdom, they are not so progressive and, therefore, weigh less heavily on the higher managerial incomes. Contrary to popular assertions, German exporters gain little, if anything, from more favourable treatment than that accorded to exporters in this country, and this is particularly true since the abolition of the German taxation export incentives at the end of last year.

As far as the availability of capital is concerned, the United Kingdom exporter can draw on the resources of the banking system and the London money market, which can normally provide adequate credit for most classes of business, and in this respect he still has an advantage over his German competitor, who not only has more difficulty in raising the money for financing both production and export, but has to pay higher rates of interest. On the question of export credit insurance, there is no evidence that the U.K. exporter with access to E.C.G.D. facilities is in any way at a disadvantage compared with his German counterpart.

There is little doubt that the pull of the home market in relation to output available has been stronger in the United Kingdom than in Germany during the last 18 months. While demand grew quite rapidly in both countries in 1955, Germany was able to expand her output (and her imports) sufficiently to satisfy all the extra demand without running into balance of payments difficulties, and without substantial price increases, while the United Kingdom was unable to do so. Being unable to meet the whole demand, many U.K. manufacturers have preferred to sell their goods at home where this is easier. In marked contrast, German sales efforts in overseas markets have been quite exceptionally vigorous, stimulated by a determination to rebuild the economy and restore Germany's power and prestige abroad.

If the challenge of German competition is to be success-

* Soviet [illegible] countries excluded from calculations.
† 28th J[illegible] 1956.

fully met, British industry must pay special attention to improving its sales promotion abroad. At the same time, it must invest more in the modernisation and expansion of industrial capacity in this country. In the long run, a competitive advantage will rest with the country which is most successful in preventing inflation, and which achieves the most rapid increase in efficiency and sales promotion, and advances in technology.

Personal News

THE ENGLISH ELECTRIC COMPANY announces that SIR GEORGE H. NELSON, BT., Chairman and Managing Director of the Company, has relinquished the position of Managing Director in order to devote the whole of his time to the duties of Executive Chairman of the Company. The Board has appointed MR. H. G. NELSON, the Deputy Managing Director, to be Managing Director as from July 1st, 1956.

UNITED COKE AND CHEMICALS CO., LTD., of Treeton, Rotherham, a branch of The United Steel Cos., Ltd., announce that MR. P. H. PINCHBECK has been appointed Technical Assistant to the Commercial Manager (Mr. R. T. Hayes). For the past eight years, Mr. Pinchbeck has been employed as a Senior Chemist, in charge of research into the oxidation of certain hydrocarbons, with the Coal Tar Research Association at Gomersal, near Leeds.

MR. W. T. COOPER, who has been Editor of the Journal and other publications of The Institute of Fuel since 1948, has resigned to take up an appointment as Senior Research Officer, Australian Commonwealth Scientific and Industrial Research Organisation. Mr. Cooper will be in charge of information and public relations work in the Coal Research Section of C.S.I.R.O. at North Ryde, Sydney, N.S.W. He is to succeed Mr. N. Y. Kirov as Honorary Secretary of the Australian Membership of The Institute of Fuel in January, 1957.

ON reaching retiring age MR. E. J. CLARKE, Chief Engineer of Murex Welding Processes, Ltd., Waltham Cross, Herts, has retired after 38 years service with the Company, and to mark the occasion Mr. J. M. Willey, Director and General Manager of the Company, presented Mr. Clarke with a fitted wardrobe on behalf of Murex employees.

NORTHERN ALUMINIUM CO., LTD., announce that MR. A. K. JORDAN, Assistant General Sales Manager, has left the Company to take up the post of General Sales Manager with Aluminum Co. of Canada, Ltd. (Alcan). Mr. Jordan was Alcan's Export Sales Manager before coming to Northern Aluminium in 1951, and has been with the Aluminium, Ltd. group for 26 years. He is succeeded by MR. J. H. MAYES, who joined the Technical Department of the Company in 1934. Since 1946, Mr. Mayes has successively been responsible for Northern's Sales Development, Castings and Forgings Sales and, latterly, Export Sales Departments.

METROPOLITAN-VICKERS ELECTRICAL CO., LTD., announce that MR. A. C. ELLIS, who became District Manager of Cardiff Office in 1952 and before that date had been attached for many years to the Company's Manchester Office, has now returned there as District Manager. His appointment is consequent on the retirement of MR. J. B. HARTLEY, who will remain in an advisory capacity until the end of September. MR. J. S. HALL, who was recently appointed Assistant Manager, Cardiff Office, has now been appointed Manager, in succession to Mr. Ellis.

A.P.V.-PARAMOUNT, LTD., stainless and alloy steel founders, announce the appointment of MR. E. J. COOPER, as Production Superintendent, in succession to MR. L. J. L. DAVIES, who has joined the Company's Sales Staff as Southern Area representative.

HENRY WIGGIN & CO., LTD., producers of specialised high nickel alloys in Birmingham, Glasgow and Hereford, announce the appointment of MR. H. W. G. HIGNETT as Assistant Managing Director. Mr. Hignett, who is a Director of the Company, was responsible for technical (metallurgical) control and development in all Wiggin plants. He was previously Superintendent of the Mond Nickel Company's Birmingham Research Laboratory.

MR. E. W. HANCOCK, Director and General Manager of Humber, Ltd., the manufacturing organisation of the Rootes Group, has been elected President of the Institution of Production Engineers, and MR. H. G. GREGORY, Manager of the Northern Factories of The Fairey Aviation Co., Ltd., has been elected Chairman of Council.

Automatic Controls Pioneer Visits U.K.

OVER fifty years ago, Mark C. Honeywell started a small business in Wabash (U.S.A.) to make and sell "comfort"—in the form of automatic temperature controls, or thermostats. In 1927, he merged his Company with another of similar type to form the Minneapolis-Honeywell Regulator Company. A recent visit of Mr. Honeywell to his British associates—Honeywell-Brown, Ltd., coincided with the end of a two-year development programme during which the Lanarkshire factory has more than doubled its output.

The location of the factory was a result of post-war economic policy for Scotland—the introduction of new industries. The success of this policy has been heavily underlined by the standards of precision engineering now confidently associated with the automatic controls produced in Lanarkshire. During the past two years, the number of employees has doubled, and more employment opportunities will result from an extension of the manufacturing area now under construction.

The factory extension has been made necessary by continued acceleration of the demand for all products. And the demand has not been limited to home markets: last year the Company exported over 40% of its output to Europe, North America, and other overseas markets.

Jamaica Alumina Expansion

PLANS for the construction of a second alumina manufacturing plant in the island of Jamaica to utilise local bauxite ores and keep pace with expanding primary aluminium capacity in Canada have been announced by Aluminium, Ltd. The Company's subsidiary, Alumina Jamaica, Ltd., will start construction immediately on a new plant to produce 245,000 tons of alumina per year. First production is expected by mid-1958, at an estimated capital cost of approximately $35,000,000. The Company's initial plant is now being extended to 550,000 tons of alumina per annum and, with the new plant, is designed principally to support the 330,000 tons of primary aluminium capacity planned for the Company's Kitimat (British Columbia) smelter.

Magnetic Measurements

By C. E. Webb, B.Sc.(Eng.), A.M.I.E.E.

(from the National Physical Laboratory)

The basic principles of the chief methods of determining the properties of ferromagnetic materials—ballistic, slowly varying flux, and alternating current methods—are described. Special consideration is given to the testing of permanent magnets by standard and comparative methods, and to the conditions necessary to ensure that test results give reliable indications of practical performance. Some representative methods of measuring field-strength are reviewed, and in conclusion the accuracy attainable in magnetic measurements is briefly discussed. This article is based on a lecture delivered to the Permanent Magnetic Association in Sheffield on May 19th, 1954.

1. Introduction

A LARGE proportion of all metallurgical products possess the property generally described as "being magnetic," or—more precisely—are ferromagnetic materials. Moreover, a considerable fraction of these materials is manufactured for purposes for which the magnetic properties are of the first importance.

A broad survey of the chief methods employed to measure the properties of ferromagnetic materials may, therefore, be of interest to metallurgists in general, and to those concerned with the production of permanent magnets in particular. It is proposed to deal mainly with the general principles of the methods, referring to details of individual examples only for illustration, and to consider them primarily in their purely magnetic aspects.

2. Quantities to be Measured

The properties of ferromagnetic materials are in the main expressed in terms of two quantities—the magnetising force, or magnetic field strength, H, to which the material is subjected, and the resulting magnetic flux density, or induction, B, produced in it. The relation between these two quantities is a complex one which can be represented only graphically by the familiar magnetisation curve and hysteresis loop, examples of which are shown in heavy lines in Fig. 1.

The magnetisation curve gives the variation of B as H is increased progressively from zero, while the hysteresis loop gives its variation as H is varied cyclically between equal positive and negative values, H_{max}. Magnitudes associated with these curves, which are of practical importance, are : —

(1) Permeability, μ, given at any point on the magnetisation curve, such as X (Fig. 1), by the ratio B/H, i.e., by the tangent of the angle of inclination of the line joining X to the origin. If, at any point on the magnetisation curve or hysteresis loop, the progressive variation of H is halted and a change $\triangle H$ is made in the reverse direction, the ratio of the resulting change $\triangle B$ to $\triangle H$ is called the "incremental permeability," $\mu_\triangle$, or—if the reversal occurs at a point on the descending limb of the hysteresis loop—the "recoil permeability."

(2) Area enclosed by the loop, which is a measure of the energy expended in carrying unit volume of the material through the cycle of magnetisation—the hysteresis loss.

(3) Value of B when H is reduced from H_{max} to zero—the remanent flux density—and particularly the limiting value approached by this quantity as H_{max} is indefinitely increased, which is known as the remanence, B_r.

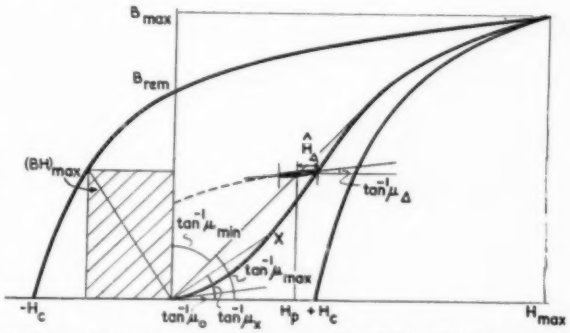

Fig. 1.—Magnitudes associated with the magnetisation curve and hysteresis loop.

(4) Value of demagnetising H required to reduce B to zero—the coercive force—and particularly the limiting value approached by this quantity as H_{max} is indefinitely increased, which is known as the coercivity, H_c.

(5) Maximum numerical value of the product (BH) for points on the portion of the loop—generally known as the demagnetisation curve—between the remanence and coercivity points. This quantity is represented graphically by the area of the shaded rectangle in Fig. 1.

Of these five quantities, (1) and (2) are important chiefly in connection with soft magnetic materials : (3), (4) and (5) are chiefly significant in connection with permanent magnet materials.

Although the magnetisation curve and hysteresis loop have complex shapes, they appear at first sight to give a definite and clear-cut representation of the relation between B and H. In practice, however, complications are found which necessitate precise control of the conditions of measurement if unambiguous and reproducible results are to be obtained. For example, attention must be given to :—

(*a*) Effect of previous magnetic history. To avoid variability in results from this cause, materials must be demagnetised before testing by applying a strong field and reversing it repeatedly while gradually reducing it to zero.

(*b*) Method of magnetisation. Even after demagnetisation, the value of B measured at a given value of H is found to depend on the way the value of H is approached. The most reproducible relationship between H and B is obtained if the material is maintained in a symmetrically cyclic magnetic (s.c.m.) state, i.e., the

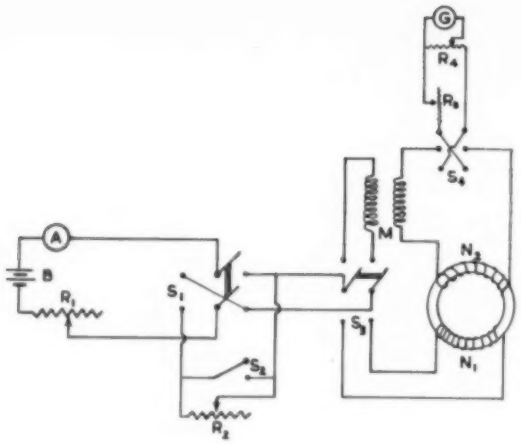

Fig. 2.—Circuit for ballistic test (on ring specimen).

value of H alternates between equal positive and negative values.

(*c*) Accommodation. In many materials, even in the s.c.m. state, the value of B at a given value of H increases with repeated cycles, and only becomes constant after a considerable number of cycles have been carried out. This effect is known as " magnetic accommodation," and, to obtain the most reproducible results, measurements should be made in the s.c.m. condition corresponding to complete accommodation.

An important matter in connection with measurements of any kind is the system of units to be employed. In electrical work the m.k.s. (metre-kilogram-second) system embodying all the practical electrical units, has advantages and is being increasingly adopted. The magnetic units in this system, however, are less familiar and convenient, and since—particularly in dealing with permanent magnet materials—it has been almost universal practice to use c.g.s. units, it is proposed to express in these units any magnetic magnitudes which it may be necessary to introduce. The c.g.s. units of H and B are respectively the oersted and the gauss, and as this system is based on the assumption that the permeability of a vacuum (and hence, very approximately, of air) is unity, the flux density in gauss produced in air or other " non-magnetic " medium by a field may be taken to be the strength of the field in oersteds.

3. The Magnetic Circuit

A useful approach to the basic principles of the chief methods of testing magnetic materials is by way of an analogy between magnetic and electrical magnitudes, leading to the conception of the Magnetic Circuit. The resemblance between the magnetic and the electric circuits, is, however, purely formal, since in the magnetic circuit there is no actual flow—and hence no continuous expenditure of energy—as there is with an electric current.

The magnetomotive force, F, in a magnetic circuit, measured by the ampere-turns operating in it, may nevertheless be regarded as driving the total flux, Φ, through the reluctance, Z, of the circuit, giving a magnetic equivalent to Ohm's law, which may be written—if consistent units are employed—

$$F = \Phi Z$$

The reluctance of any uniform portion of the magnetic circuit is derivable from its dimensions and permeability by means of the expression $l/\mu A$, which corresponds exactly to the expression for electrical resistance with the reciprocal of permeability—or " reluctivity "—replacing resistivity. The inverse of reluctance, known as " permeance " is conveniently used when dealing with parallel magnetic paths, just as " conductance " is with parallel electric circuits.

The magnetic Ohm's law may be applied, like its electrical counterpart, to a portion of a circuit, and makes it possible to deduce the " magnetic potential difference " across it from its reluctance and the total flux in it.

4. Methods of Measurement

4a. General

By comparison with measurements of current and voltage, measurements of the magnetic quantities B and H suffer from two considerable difficulties :—

(1) The inaccessibility of B and H in magnetic materials to direct measurement. The only method of measuring B is by integration of the e.m.f. induced, in a search-coil surrounding the material, by the change of linkage resulting from movement of the coil or variation in the flux : such a method can only yield the mean value of B over the cross-section. H is even less accessible, and can in general be derived only from measurements in an adjacent air-space, in conditions which make it legitimate to assume that the value so measured is also the value of H inside the material.

(2) The absence of an effective magnetic insulator. The reluctivity of air is not large enough, compared to that of magnetic materials generally, to ensure that the flux is strictly confined to the ostensible magnetic circuit, and magnetic potential differences between various points in the circuit produce leakage fluxes which may make the effective magnetic circuit hard to define. The occurrence of such leakage fluxes is a factor which often makes it preferable to treat magnetic flux paths on the basis of permeance rather than reluctance.

These two difficulties in combination make it essential for precise measurement to achieve an approximation, at least, to the condition of a perfectly uniform magnetic circuit.

4b. Tests on Rings

A uniformly wound, circular ring of magnetic material provides one of the simplest and most satisfactory means of attaining a uniform magnetic circuit. Provided its cross-section and magnetic properties are uniform around the circumference, the magnetomotive force and reluctance of any portion of the circuit are directly proportional to its length. Considerations of symmetry then allow H to be calculated from the total magnetising ampere-turns and the mean length of the magnetic circuit. Values of B are derived by integration of the e.m.f. induced in a secondary winding, or search-coil, when changes in the magnetisation are made. The integration of the induced e.m.f. is usually carried out by means of a ballistic galvanometer, i.e., by the ballistic method which forms the basis of most standard methods of magnetic testing.[1] The circuit for applying this method to a ring is shown in Fig. 2. Its operation is too familiar to need detailed description, but it may be pointed out that, with switch S_3 closed in the downward position and S_2 closed, the ring may be magnetised to

any desired value of H by suitably adjusting R_1, and may be put into an s.c.m. state by reversing S_1 repeatedly. H can then be deduced from the current read on the ammeter, A, while B is derived from the ballistic galvanometer deflection when the magnetisation is reversed. The galvanometer is calibrated in the same circuit conditions by closing S_3 in the upward position and reversing a known current in the primary of a mutual inductor, M, of known inductance. By opening S_2, an additional adjustable resistance, R_2, is inserted, allowing any desired point to be obtained on the hysteresis loop, whose symmetrical tip-points are determined by the setting of R_1. Since magnetic changes often require appreciable time to complete themselves, a ballistic galvanometer of long period—not less than 10 seconds—is desirable.

Owing to the simple conditions provided by the ring test, it has been largely used as a standard method. Its only inherent inaccuracy arises from the radial non-uniformity of magnetisation. To minimise the effect of this non-uniformity, the ratio of radial width to mean diameter should be kept small—preferably not greater than $\frac{1}{4}$. The chief limitations of the ring-test are :—

(1) The practical inconvenience of having to wind both search-coil and magnetising coil on each specimen. The number of turns is usually large only in the magnetising coil, and the labour of winding these may be minimised, either by using a multi-strand cable with a plug and socket connector to connect the strands in series, or by means of a system of conductors provided with movable links or switches, which can be opened to insert the ring and then closed to connect the conductors in series.[2]

(2) Directional properties cannot be tested, except in the special case of spirally wound strip, in which the flux is in the longitudinal direction at every point. Such a spiral core can be regarded as a uniform ring having a distributed air-gap of effective length $tt'/2\pi r$, where t and t' are the thicknesses of the strip and of the gap between successive turns of the spiral, and r is the mean radius of the ring.

4c. Tests on Straight Samples—Yoke-Permeameters

To avoid the limitations of the ring-test, methods of testing straight bars or strips have been developed, the most important of which are the various forms of yoke-permeameter. In these, the necessity for winding large numbers of turns on the test-piece is obviated by using permanently made-up coils and threading the magnetic

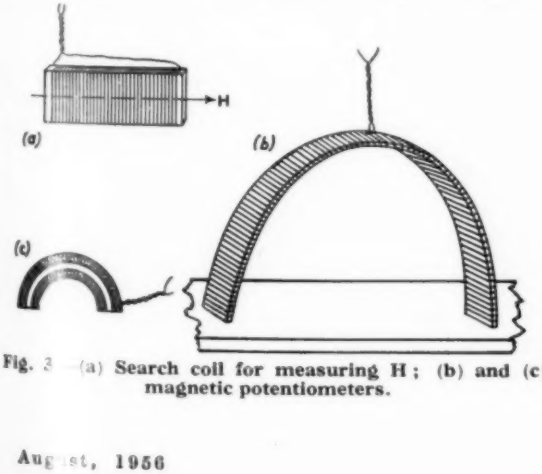

Fig. 3.—(a) Search coil for measuring H; (b) and (c) magnetic potentiometers.

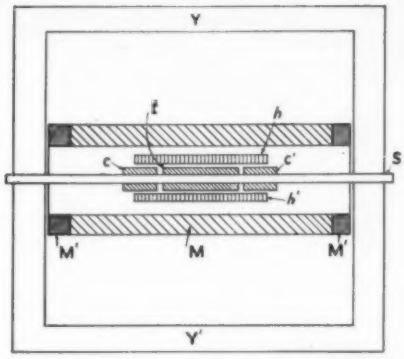

Fig. 4.—Diagram of N. P. L. permeameter.

circuit through them. This involves a non-uniform magnetic circuit comprising, besides the test-piece, one or more yokes and the joints between the yokes and the test-piece. For such a circuit, H cannot in general be calculated from the magnetising ampere-turns, but must be measured in the air-space adjacent to the test-piece by means of search-coils wound on a non-magnetic core (see Fig. 3*a*) placed with their axes parallel to the direction of the field. These H-search-coils, if connected to the ballistic galvanometer, can be used in exactly the same way as the B-search-coil, to measure the flux density in the air—or, strictly speaking, in the non-magnetic core on which they are wound—close to the surface of the test-piece. The flux density in a non-magnetic medium, however, provides an accurate measure of the field-strength, and thus a ballistic measurement with suitably placed H-search-coils gives the value of H in the test-piece, provided the conditions are such that there is no gradient of H perpendicular to the surface.

An alternative to H-search-coils is a magnetic potentiometer.[3,4] This instrument (see Fig. 3*b* and 3*c*) consists of a uniformly-wound helix which, if connected to a ballistic galvanometer and treated as a search-coil, measures the magnetic potential difference between the end-surfaces of the helix. It is particularly useful for measuring the magnetic p.d. across irregular portions of a magnetic circuit, e.g., joints, and can be made to give the mean value of H along the length of a test-piece to which it is applied.

A typical form of yoke permeameter is that used at the National Physical Laboratory,[5] shown diagrammatically in Fig. 4. In this instrument the magnetic circuit of the specimen, S, is completed by two rectangular yokes, Y and Y', of large cross-section, and having a large area of contact with the specimen. The reluctance of the return path for the flux, thus provided, though small, is not negligible, and besides the main magnetising winding, M, additional magnetising windings, M', are employed at the ends of the specimen to apply "compensating" m.m.f.'s which balance the magnetic p.d. across the reluctance of the yokes and joints. The current in these windings is adjusted for each reading to secure uniform magnetisation along the test-length covered by the search-coils. The uniformity of magnetisation is indicated by zero deflection on a ballistic galvanometer connected to the central B-search-coil, t, and end search-coils, c and c'—each having half the number of turns of t—in series opposition, when the main and compensating magnetising currents are simultaneously reversed. In this condition the readings of the H-search-coils, h and

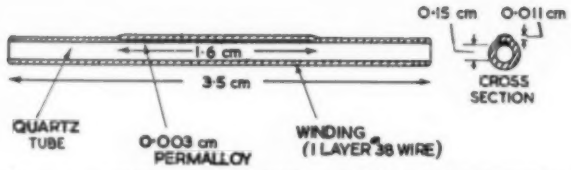

Fig. 5.—Construction of high sensitivity H-measuring element (Carr).

h', are independent, over a wide range, of their distance from the surface of the specimen, and thus give true indications of the value of H in the specimen : in any other condition, in which appreciable flux leakage occurs between the middle and end of the test-length, the apparent value of H varies with the distance of the H-coils from the surface at a rate approximately proportional to the magnitude of the flux leakage.

This observation suggests that an alternative criterion of uniform magnetisation along the test-length is radial constancy of the H-search-coil reading. Further, in this condition, the value of H should be calculable from the ampere-turns/cm. of the main magnetising winding, and it has in fact been found[2] that agreement between this calculated value and the value measured by H-search-coils is a simple and satisfactory indication that the compensated, or uniformly magnetised, condition has been established.

A wide variety of yoke permeameters has been devised, but it seems probable that the general arrangement shown in Fig. 4 is one of the most satisfactory—if only because it has been independently invented so frequently. For example, a permeameter described by Sanford[6] employs the same general principles, though with the following modifications in manipulative details :—

(1) The compensating m.m.f. is provided by four coils placed on the yokes, connected in series with the main magnetising coil, the number of turns in the coils being fixed to give the best compensation on the average for different specimens and values of H.

(2) Since exact uniformity is not obtained, the radial variation of H is determined for each reading by means of H-search-coils at two different distances from the specimen, and the value of H at the surface of the specimen is deduced by extrapolation from these two readings.

(3) Flip-coils, rotatable through 180°, are used to measure H statically instead of deducing it from the effects of reversing or otherwise varying it. The static measurement is particularly valuable in determining small values of H when obtaining hysteresis loops, since in the dynamic method such large values can only be derived as a small difference between two large, directly measured quantities.

There have been interesting recent developments, connected with yoke permeameters, in methods of measuring H and of applying a cyclically varying magnetising field. For the measurement of H, a direct-reading magnetometer has been devised[2] in which a Silmanal* permanent magnet, approximately 2 mm. long, mounted on a spring-controlled pivoted spindle or —for greater sensitivity—a short suspension, is deflected by the field to be measured. The Silmanal magnetometer, compared with the combination of search-coil and ballistic galvanometer, greatly simplifies the measurement of H, but naturally involves some sacrifice of accuracy.

A serious limitation to the use of search-coils for measuring H is the lack of sensitivity in weak fields. Magnetometers operating on the saturable reactor principle have, however, been developed,[7,8,9] which allow fields down to 10^{-3} oersted to be measured. The principle has been applied in various ways, but the basis of all the methods is that in a Permalloy or Mumetal wire, subjected to a high frequency (say 5 kc./s.) magnetisation, fluxes of the second and other even harmonic frequencies are produced when a polarising field is present. Thus, if the wire is placed with its length parallel to the field to be measured, and an adjustable opposing field is applied by means of a small, uniformly wound solenoid around the wire, the disappearance of the even harmonic fluxes provides a sensitive indication of balance between the two fields, and the strength of the unknown field is given by the value of the solenoid field, calculated from the number of turns/cm. and the current through it. The arrangement of one form of magnetometer employing this principle is shown in Fig. 5. The transverse dimensions are made small so that the wire can be placed close to the surface of the test-piece without exerting a significant influence on the field in the test-piece. In the instrument shown, it will be seen that the distance from the surface of the test-piece is only about 0·011 cm. The application of this method of H-measurement is limited by the heating, due to the solenoid current, to fields not greater than about 50 oersteds.

An ingenious device for applying a cyclically varying magnetising field, without a source of current being needed, is the Varimag permeameter,[2] in which the field is provided by a large Alcomax permanent magnet fitted with soft iron shoes. By rotating the magnet slowly through 180°, or some smaller angle, the field can be varied continuously from a positive to an equal negative value.

Permeameter methods are in general capable of giving results of high accuracy, but are slow in operation, particularly for the delineation of hysteresis loops in which complicated compensation adjustments are necessary.

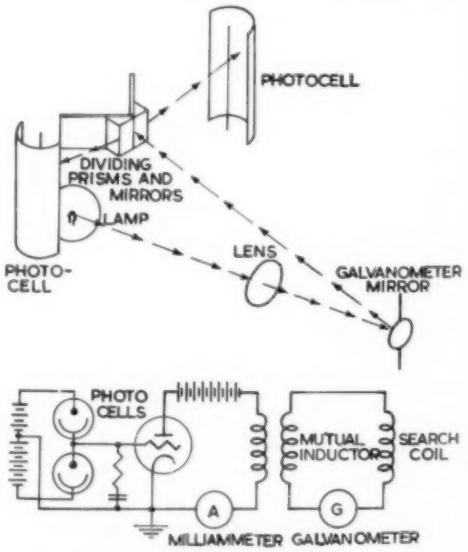

Fig. 6.—Photo-electric fluxmeter (Edgar).

* Silmanal is an alloy, resembling the well-known Heusler alloys, having an extremely high coercive force, so that the strength of the magnet is unaffected by the application of fields up to at least 3,000 oersteds.

5. Continuous Delineation of Magnetisation Curves and Loops

To obtain the relation between B and H more rapidly than is possible by the point-by-point determinations involved in ballistic tests, without resorting to a.c. methods (which are necessarily limited to laminated materials, and, owing to the effects of eddy currents, do not measure purely magnetic properties) a number of methods have been developed in which curves connecting B and H are traced continuously in a period of the order of a minute. Accurate integration of induced e.m.f.'s over a period of this magnitude presents considerable difficulties, but by using integrating amplifiers with negative feed-back, simultaneous deflections along two perpendicular ones, proportional respectively to B and H may be obtained satisfactorily.

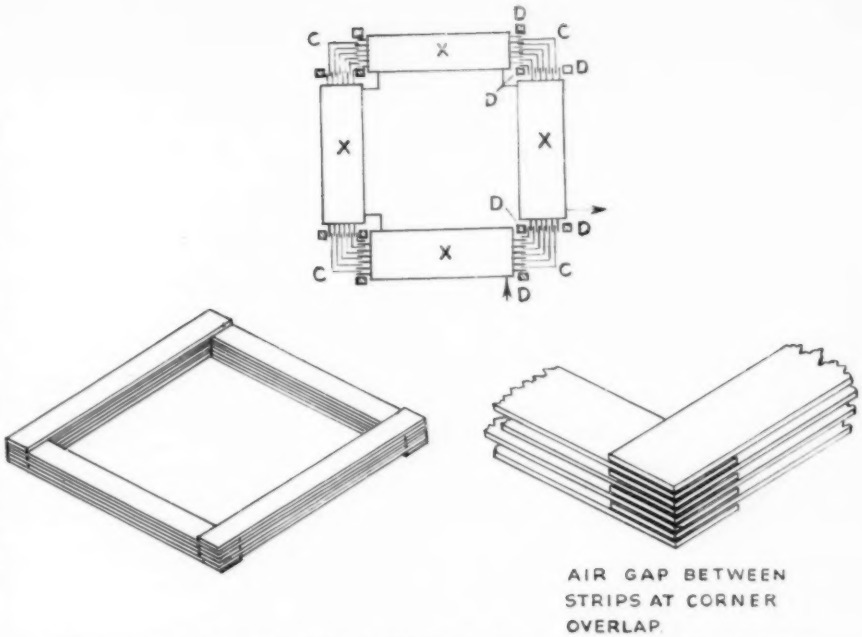

Fig. 7.—(Above) Sketch of Lloyd-Fisher square; (below) assembly of strips in Epstein square.

An instrument for carrying out the integration photo-electrically,[10] is shown in Fig. 6. A change of linkage in the search-coil produces a current in the galvanometer, G, the resulting deflection of which disturbs the balance between the two photo-electric cells. They are so connected as to vary the current in the mutual inductor primary to produce a linkage in the secondary balancing the original linkage change in the search-coil. If a voltage proportional to the primary current is fed to the plates of a cathode-ray oscillograph, a deflection proportional to the flux linking the search-coil is produced. With two such photo-electric integrators, associated respectively with H- and B-search-coils, and operating on the X and Y plates of the oscillograph, magnetisation curves and hysteresis loops may be rapidly delineated.

Other methods have been described[11,12] for automatically plotting B/H curves on a drum recorder, integrating the induced e.m.f. mechanically by means of a motor having a linear speed-voltage relation, which gives the recording pen a displacement proportional to the time-integral of the voltage applied to it.

In tests by these methods, involving a slowly varying flux, the most convenient form of sample is the ring, for which the magnetising current provides a voltage directly proportional to H. For straight samples this condition is only fulfilled if compensation is maintained, which is extremely difficult when the magnetisation is being varied continuously. Difficulty is also experienced from over-loading of the integrator when dealing with curves having a large value of dB/dH ("rectangular loop" materials), while stability requirements are very severe.

6. Alternating Current Measurements

In the large proportion of the steel used for electrical purposes, which is subjected to alternating magnetisation and must therefore be in sheet form, the most important practical measurement to be made is the determination of the total power loss due to hysteresis and eddy currents. This loss is usually measured directly by means of a wattmeter at normal power supply frequencies: at higher frequencies, a.c. bridge or resonance methods have to be employed.

Permeability measurements are generally less important than loss measurements with a.c. magnetisation, and it is indeed a matter of some difficulty to decide what is the best definition of permeability to adopt under alternating conditions. The ratio B_{max}/H_{max} provides the closest analogy to the d.c. permeability determined in the s.c.m. state, but H_{max} and B_{max} are not in general simultaneously occurring values, and, furthermore, are not directly derivable from the effective, or r.m.s., values of magnetising current and induced voltage. In this, as in a.c. magnetic measurements generally, allowance must be made for the effects of waveshape.

To discuss the general problems of a.c. magnetic measurements would require more space than is available, and it is proposed only to note the way in which the principles discussed in connection with d.c. tests are applied in a.c. measurements.

The necessity for an approximation to a uniform magnetic circuit holds in the a.c., as in the d.c., case. Ring samples thus again provide theoretically the most suitable form of test-piece. To avoid their practical drawbacks, however, built-up magnetic circuits, composed almost entirely of the test-material, are generally employed in the form of either the Lloyd-Fisher[13] or the Epstein[14] square. In each of these assemblies four bundles of strips are placed inside bobbins carrying magnetising and search-coil windings so as to form a closed magnetic circuit in the shape of a square. The joints at the corners are made of low reluctance—in the Lloyd-Fisher square by interleaving small corner-pieces,

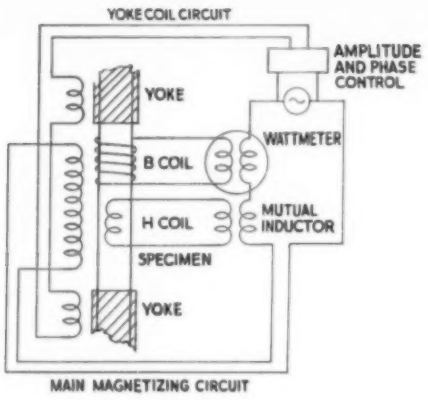

Fig. 8.—A.C. yoke tester (Schenk).

bent at right angles, between the strips (see Fig. 7, upper diagram); in the Epstein square by interleaving the strips themselves (see Fig. 7, lower diagram). The reluctance of the joints, though small, is not entirely negligible, but its effect on the apparent permeability is not important (if desired an approximate empirical correction can be made for it), while its effect on the losses, which if considerable would be more serious, is very small. Either of these assemblies may thus be regarded as forming a ring sample without the necessity for winding turns on each test-piece. If the properties of the sheet differ in different directions relative to the direction of rolling, the strips may all be cut in the direction in which the properties are to be measured, or, if an average is required of the properties in all directions, a satisfactory approximation may be obtained by cutting half parallel and half perpendicular to the direction of rolling.

Methods analogous to the d.c. compensated yoke permeameter (see section 4c and Fig. 4) have also been developed though not widely used. A diagrammatic representation of such a method[15] is shown in Fig. 8. In the a.c. case the compensating m.m.f.'s must be adjusted not only in amplitude but also in phase to produce uniform magnetisation along the test-length. As in the d.c. permeameter, it is found that the most satisfactory indication of uniformity is equality of the value of H measured by search-coil with that calculated from the ampere-turns/cm. of the main magnetising winding. This equality is readily tested by connecting a mutual inductor with primary in series with the main magnetising winding and secondary to oppose the induced e.m.f. in the H-search-coil, the appropriate value of the inductor being calculated from the number of turns/cm. of the magnetising winding and the area-turns of the H-search-coil. This method of testing has been applied to whole sheets as well as to strip-samples.

One of the main difficulties in a.c. measurements is the occurrence of large harmonic components of currents and voltages, owing to the non-linear relation between H and B. In the endeavour to avoid errors from this source, calorimetric methods of loss measurements have been employed. Such methods are in general slow and subject to radiation errors, but by the use of sensitive thermocouple detectors and thermal balance methods, accurate readings can be obtained in a few seconds. These methods, however, are only suitable for research laboratory use, and are not readily applicable to industrial routine testing.

7. Measurements on Permanent Magnets

7a. General

The chief function of a permanent magnet is normally to maintain flux in an external space, and magnetic circuit considerations show that, when doing so, the magnet itself is subject to a demagnetising field. The second quadrant of the B/H loop, or demagnetisation curve, is thus of special interest in connection with permanent magnet materials.

At any point on the demagnetisation curve the product (BH), it may readily be shown, is proportional to the external energy available per cu. cm. of the material. This quantity is, therefore, known as the "energy product," and its maximum value, $(BH)_{max}$, which indicates the maximum external energy that a cu. cm. of the material can provide, is an important characteristic of a permanent magnet material. A simple means of reading the energy product directly from the demagnetisation curve has been described,[16] in which the curve is plotted on specially prepared graph-paper carrying a set of hyperbolae corresponding to a series of values of (BH), as shown in Fig. 9. The energy product at any point on the demagnetisation curve is shown by the hyperbola passing through it, and the value of $(BH)_{max}$ is given by the hyperbola tangential to the demagnetisation curve (interpolating, if necessary, between the two nearest hyperbolae plotted).

The magnetic condition of the material of a given permanent magnet operating under specified conditions can be found graphically by means of two sets of curves :—

(1) Lines representing the magnetic properties of the material, consisting of the demagnetisation curve (B_cH_c, Fig. 9) and "recoil" lines (such as $a\ e\ c$ and $f\ h\ g$, Fig. 9) which show the variation of B as the negative, or demagnetising, H is diminished after reaching the value required to bring the material to some definite point on the demagnetisation curve (a or f, Fig. 9).

(2) Lines determined by the geometric configuration of the magnet and its gap, comprising a series of straight lines (such as Oa and Oe in Fig. 9) drawn from the origin in the second quadrant. Each of these lines represents the various possible magnetic conditions of the permanent magnet material for a particular configuration of the magnetic circuit, and its slope, B/H, is given by the ratio of the total external permeance to the permeance of the volume occupied by the magnet for that configuration. This ratio is known as the "permeance coefficient," P, of the magnet.

Thus, if a magnet has a constant permeance coefficient, P_1, corresponding to the line Oha (Fig. 9), its condition will be represented by the interesection of Oha with the demagnetisation curve, i.e., by the point a. If, however, the external permeance is increased by introducing soft iron poles or an armature into the magnetic circuit, so that the permeance coefficient is increased to P_2, corresponding to the line Oe, the magnetic condition of the material will be represented by the point e at which Oe intersects the recoil line $a\ e\ c$ from the point a on the demagnetisation curve. Similarly, if it is desired to use the magnet in the configuration giving the permeance coefficient P_1 to maintain a constant flux, even if liable

to be subjected to external demagnetising fields, it may be stabilised by applying a demagnetising field H_s, bringing the material to the condition represented by the point f. After the removal of H_s the material recoils to the point h at which the recoil line $f\ h\ g$ is intersected by the line Oha corresponding to the permeance coefficient P_1. Any subsequent temporary application of a demagnetising field (provided it does not exceed H_s) will merely make the material change its condition along the line fh so that after removal of the demagnetising field it will return to the point h.

It is clear from the foregoing considerations that the performance of a magnet with a given configuration depends on its magnetic history subsequent to magnetisation, and that if useful measurements are to be made to determine the relative suitability of different materials for a particular purpose, they must be carried out under conditions accurately reproducing those in which the materials will be required to operate. (In the preceding discussion the position has been simplified by ignoring the variation in the magnetic condition of the material which normally occurs from point to point in a permanent magnet. This simplification involves no difference of principle and does not affect the practical conclusion drawn).

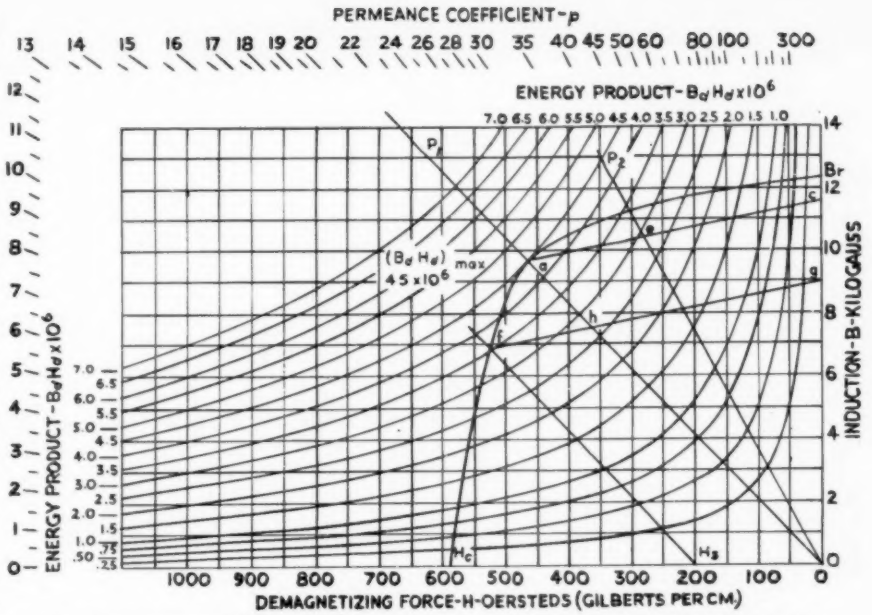

Fig. 9.—Demagnetisation curve with recoil and permeance lines and hyperbolae for direct reading of energy products (Maynard and Mitch).

7b. Application of Permeameter Methods

The permeameter methods of measurement described in 4c, provide the most accurate means of determining the properties of permanent magnet materials, and may be employed with only slight modifications in detail, provided bars or blocks of uniform cross-section are available. For example, it is difficult to apply compensation to the short samples usually employed in testing permanent magnet materials, but owing to their relatively small effective permeability the resulting error is often negligible. If desired, a correction can be made for the effect of non-uniform magnetisation by using two sets of H-search-coils at different distances from the surface of the sample and extrapolating from the two readings to obtain the value of H at the surface, as described in connection with Sanford's permeameter (4c).

An alternative form of magnetic circuit, which is particularly suitable for testing high coercivity alloys in the shape of short blocks, consists of an "isthmus" type of electromagnet[4] with the sample placed between large pole-pieces in the gap.

7c. Other Standard Methods

Permeameter methods, though giving the highest absolute accuracy and providing fundamental data for the magnet designer, are too slow for routine measurements, and alternative methods have been developed for obtaining information rapidly on the properties of materials, not only in the form of uniform straight samples, but also in the simpler shapes of finished magnet.

Two methods are recognised by the British Standards Institution as commercial standards.[17] In each a magnetic circuit containing a narrow gap is energised by the test-magnet, which can be magnetised and demagnetised to any desired degree by means of a suitable coil, the current in which provides a measure of H. In one method B is deduced from the deflection of a moving-coil mounted in the air-gap and carrying a known current—essentially a moving-coil galvanometer operating inversely. In the second, and generally adopted, method B is derived from the e.m.f. induced between the copper rim and spindle of a mild steel disc rotating at a known speed in the air-gap. The circuit constants are adjusted to provide direct readings of H and B, and corrections are applied for the reluctance of gap, pole-pieces and joints, determined by magnetic potentiometer measurements. Fair agreement with permeameter results is obtained if the B.S. procedure is adopted, but it has been shown[18] that a further correction for leakage flux not passing through the gap—and hence not affecting the B-measuring element—gives still better agreement even on very short specimens.

7d. Comparative Measurements

For acceptance testing of a series of nominally identical magnets, comparative measurements at a single condition of magnetisation are generally sufficient. The B.S. test apparatus, just described, can be used for this purpose but, in many cases, is more elaborate than necessary, and a simple comparator, operating as nearly as possible in the same way as the magnet in use, often provides the most satisfactory test. For example, tests on tractive

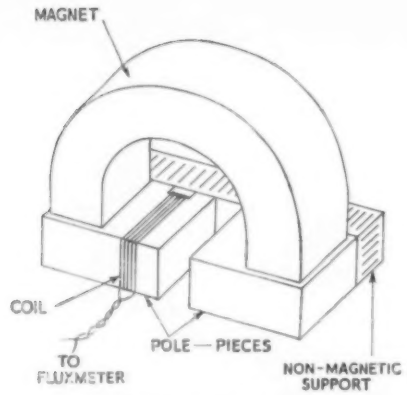

Fig. 10.—Diagram of test assembly for permanent magnets providing adjustable circuit conditions (Maynard and Mitch).

force or on the operation of a meter movement may be the best means of testing the appropriate types of magnet.

The most generally used comparative test, however, is the measurement of gap flux density. This may be carried out most simply by withdrawing or reversing a search-coil—connected to a ballistic galvanometer or fluxmeter—placed in the air-gap, but a number of alternative methods are described in the next section. To ensure that such tests give satisfactory indication of the comparative performance of different magnets in actual working conditions, attention must be paid to the following points :—

(1) The whole magnet must be magnetised to saturation before testing. This requires not merely the application of sufficient ampere-turns, but their application in such a way that the necessary magnetising field is produced at every point in the magnet. Difficulty in attaining this condition is particularly liable to be experienced in magnetising a magnet with long limbs from the ends of the limbs.

(2) As pointed out in 7*a* the test must be made with the magnet in a circuit equivalent to the assembly in which it is designed to operate. Fig. 10 shows diagrammatically a set-up in which, by adjusting the pole-pieces, the flux in the magnet can be made the same as in the working assembly. This equality may be tested by means of a search-coil wound on the magnet, and, once it is established, the position of the pole-pieces may be fixed. Comparative tests on different materials in the required magnetic circuit conditions can then be made by measuring either the gap flux density or the flux in one of the pole-pieces—using a search-coil as shown in Fig. 10 and removing the magnet from the pole-pieces—since for a given circuit configuration either of these measurements gives results proportional to the flux in the magnet. Further, if the magnet in use will be subjected after magnetisation to variation of the permeance coefficient of its magnetic circuit, or to a stabilising demagnetising field, it is essential, as explained in 7*a* that it shall be subjected to the same treatment before testing, i.e., it must be tested at a point, corresponding to the permeance coefficient of the final assembly, on the recoil line from the lowest point on the demagnetisation curve to which the material is carried after magnetisation. The misleading results which may follow neglect of this requirement can be seen from Fig. 11, which shows the demagnetisation curves of two materials I and II. If the circuit in which the magnet is to work has a permeance coefficient represented by OP_1, and the magnets are tested in the set-up of Fig. 10 at the corresponding setting, the readings on I and II will be taken at points A and B respectively, and I will appear stronger than II. If, however, in actual use the magnet is first placed in a circuit of permeance coefficient represented by OP_2, the operating conditions of I and II will be represented by points A' and B', respectively, and II will in practice be stronger than I.

8. Field Strength Measurements

Measurements of flux density in air, as has been pointed out, are important in magnet testing, but they are also required as a means of determining field strength in conditions varying widely in respect of strength and uniformity of field and the volume within which the field-strength is to be determined. Besides the ballistic method, based on the withdrawal or reversal of a search-coil, and the moving-coil and rotating-disc methods employed in the B.S. permanent magnet testers (section 7*c*), a large number of other methods have been employed, of which a few of the most important and distinctive will be briefly mentioned.

(1) Rotating coil. A widely used method consists in rotating a search-coil on a spindle, placed perpendicular to the field and driven by a synchronous motor. The voltage generated is applied by means of slip-rings to a sensitive measuring instrument, e.g., an amplifier and moving-coil meter, which may be calibrated in terms of field-strength by observations on known fields. By using very small coils the space-variation of the field can be explored even when there are large gradients. In a recent example of this type of apparatus,[19] a coil 1·5 mm. long and 1 mm. in external diameter was rotated at 3,000 r.p.m. and gave, at the maximum sensitivity, full-scale deflection for a flux density of 3 gauss.

One of the main difficulties of this arrangement is the disturbance liable to be caused by the sliding contacts at the slip-rings. To avoid this source of trouble, an air-cored transformer has been used,[20] the primary of which rotates with the search-coil, inducing in the stationary secondary an e.m.f. which may be made to give a direct reading of the field-strength on a suitable voltage-measuring instrument.

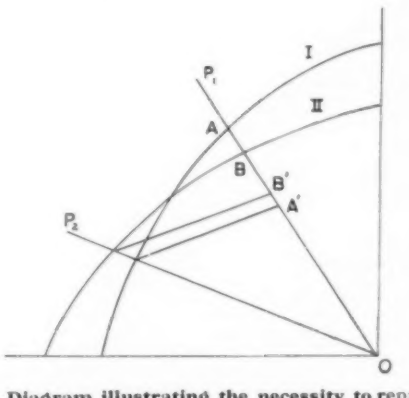

Fig. 11.—Diagram illustrating the necessity to reproduce operating conditions in comparative testing of permanent magnets.

(2) Oscillating coil. As with a rotating coil, the e.m.f. induced in a coil oscillating at a definite frequency with a known small amplitude may be used to provide a measure of field-strength in certain circumstances, e.g., in the annular gap of a loud-speaker magnet.[21]

(3) Flux-ball. A method of determining the field at a point, without using coils of very small dimensions, is provided by the flux-ball.[22,23] This is a search-coil in spherical form built-up from coaxial cylindrical windings of different lengths, each having the same uniform spacing of turns along their common axis. It can be shown that such a coil is equivalent to a solid spherical coil, and hence will measure the volume-average of the field in the sphere. This volume-average, however, is equal to the field at the centre, provided the sources of the field are entirely outside the sphere. Thus, a flux-ball, if accurately constructed, can be used to measure the field at a point with high sensitivity, even if the field-gradient is large.

(4) Gaussmeter. The Silmanal magnetometer, used as a means of measuring H in yoke permeameters (see 4*c*), may also be employed as a general field-strength measuring instrument in fields ranging from a few oersteds up to about 3,000 oersteds. When used in this way it is known as a "gaussmeter."

(5) Inductance of high permeability core. Various methods, closely related to the saturable inductor type of magnetometer used to measure H in yoke permeameters (see 4*c*) have been applied in general field measurements. One form of this method[24] is based on the change of inductance of a short Permalloy or Mumetal core, measured under a.c. magnetisation, when a steady field is superposed. The secondary e.m.f. of a transformer, wound on such a core and excited at 5 kc./s., is amplified and rectified and balanced, in the absence of an applied field, against a steady rectified voltage derived from the same supply. When a steady field is applied to the core, the balance is upset, but is restored by a d.c. excitation of the primary, the magnitude of which affords a measure of the applied field.

(6) Change of resistance. The resistance-variation produced in metals by a magnetic field, though in general small, can be used for field measurement, the best results being obtained with bismuth wire wound into a spiral coil.[25] The method is limited in application to strong fields—of the order of 10^3 oersteds—and does not provide absolute measurements: each coil must be calibrated by comparison with an absolute method, e.g., the ballistic or rotating coil methods.

(7) Hall effect. When a current flows through a metal perpendicular to a magnetic field, a p.d. perpendciular to both current and field is set up. This phenomenon, known as the Hall effect, is particularly marked in germanium.[26] The p.d. produced can be used to measure strong fields, but is not accurately proportional to the field and, as with the preceding method, a calibration by an absolute method is necessary.

Of the methods of field-strength measurement so far described, none is capable, even in the most favourable circumstances, of a higher accuracy than 1 part in 1,000 (and some of them do not approach this accuracy). The following methods are capable of a considerably higher order of accuracy, and, in particular, give high relative accuracy over long periods, so that they are suitable, for example, for long-term tests on the stability of permanent magnets.

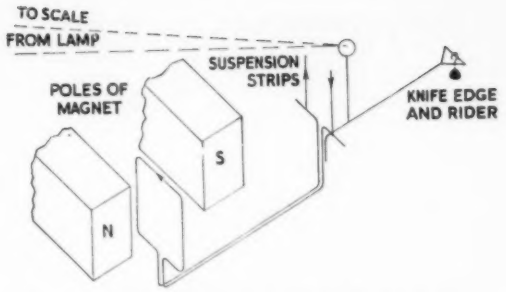

Fig. 12.—Diagram of balance for the testing stability of permanent magnets (Knight).

(8) Force on a current-carrying conductor. Since a conductor carrying a current, placed perpendicular to a magnetic field experiences a mechanical force which is proportional both to the current and the field-strength, this force may be used to measure the field-strength if the current is measured accurately by means of a potentiometer. A sensitive balance[27] embodying this principle has been developed for stability tests on permanent magnets, and is shown diagrammatically in Fig. 12. A rectangular coil, mounted on one end of the balance-arm, is fed with current through two strips on which the arm is suspended. The force exerted on the coil is balanced by the weight of a rider, precisely located on a knife-edge at the other end of the arm, indication of balance being given by an optical lever. The current required in the coil to give balance is inversely proportional to the field in which it is placed, and provides a measure of the field with a relative accuracy of 1 or 2 parts in 10^4.

(9) Null ballistic method. When a ballistic galvanometer is used deflectionally, a limit to the accuracy attainable is set by the imperfections of the instrument, e.g., in the uniformity of its field and in the elasticity of its suspension. By using it as a null instrument, errors from these sources can be eliminated, and a method has been described[28] in which the impulse applied by withdrawing a search-coil from the field to be measured is balanced by an impulse from the secondary of a mutual inductor when a current is switched on or off in its primary. The withdrawal of the search-coil and the change of current in the inductor primary are effected simultaneously by moving a lever arm, and, by careful attention to the timing of these operations, reproducibility in field measurements over long periods can be obtained within 1 part in 10^4.

(10) Proton resonance. The proton, which is the nucleus of the hydrogen atom, possesses a constant magnetic moment, M_p, associated with a constant angular momentum or spin. If a weak alternating field of frequency, f, is set up at right angles to a steady strong field, H, it is possible in suitable circumstances to detect a resonance, associated with the orientation of the proton spins in the strong field, by the energy absorption which occurs. The condition for resonance is: $f = \dfrac{2\ M_p\ H}{h}$ where h is Planck's constant. As the resonance is sharp and the frequency can be measured with great precision, H can be determined very accurately and in absolute terms, since h and M_p are natural constants known with high accuracy. A suitable form of probe has been found[29] to be a Perspex cell, containing an aqueous

solution of manganese sulphate or ferric nitrate, round which is wound the oscillator coil tuned by means of an adjustable condenser. The protons are provided by the hydrogen atoms in the water, but the paramagnetic ions introduced by the dissolved salts greatly strengthen the energy exchanges taking place and intensify the indications obtained. The method is most satisfactory for measurements in strong fields. If $H = 10{,}000$ oersteds, the resonant frequency, f, is approximately 42 Mc./s., and an accuracy of the order of 1 part in 10^5 is obtainable.

9. Accuracy

With the exception of the specially accurate methods just described, the accuracy of magnetic measurements, compared with that of other electrical measurements, is in general low. The chief sources of error are the difficulty of achieving uniform magnetisation in practically realisable circuits and the sensitivity of many magnetic materials to the effects of stress and temperature.

Even in the best conditions it is difficult to attain a higher accuracy in permeability measurements than 2 or 3 parts in 1,000, and unless considerable care is taken an accuracy of 1% is as good as can be expected. If an approximation to uniform magnetisation is not definitely aimed at, errors of $\pm$ 5% are likely, except in certain limited ranges of measurement.

Magnetic materials, however, are often so variable in their properties that even measurements of this low order of accuracy may provide all the information that is of practical significance, but where improved reproducibility in materials is attained, attention must be paid to the conditions of measurement necessary for greater accuracy if the full benefit of the improvement is to be secured.

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New Dutch Acheson Plant

ACHESON COLLOIDS, LTD., of London, manufacturers of the well-known range of "dag" dispersions, announce the incorporation of a new company in Rotterdam. Entitled Acheson Colloiden, N.V., the unit will be responsible for the production of fine dispersions at a new factory now being constructed in the Netherlands. The factory, located at Scheemda in the province of Groningen, will be completed this autumn. The Plymouth Works and London Office of Acheson Colloids, Ltd., will provide key personnel.

Arc Furnaces for Carbon Steel Ingots

As part of a major expansion scheme, to be completed by 1958/9, Round Oak Steel Works, Ltd., a member of the T.I. Group has made the bold decision to adopt electric melting furnaces to provide the increased output of steel. The first stage of the scheme will cost not far short of £1,000,000, and will include two large Birlec Lectromelt arc furnaces to be built by Birlec, Ltd., of Erdington, Birmingham.

Of particular interest is the use of electric furnaces for the first time in the United Kingdom for the production of tonnage steel for structural and other purposes. Whereas Birlec have already built a 60–70 ton furnace for special alloy steels, and have an even larger one in course of construction, the use of such furnaces for the production of plain carbon steels is an important departure from conventional practice. The furnaces at Round Oak will each have a nominal capacity of 50 tons, and together will add something like 170,000 ingot tons a year to the Round Oak output, which will then total about half a million tons annually. Technically, the Birlec Lectromelt furnaces exhibit several features of interest. Each is supplied with 20,000 kVA. of power from transformers with on-load voltage adjustment. The entire furnace roof—over 20 ft. in diameter—is lifted off and swung aside by hydraulic mechanism to permit a charge to be loaded from above in a matter of minutes.

This boldly planned scheme will be a focus of attention, not only in the Midlands, but in the steel industry generally, and marks a development of considerable economic significance.

Industrial Designers See Die Casting Techniques

A PARTY of leading industrial designers recently visited Sparklets, Ltd., the Tottenham light engineering firm. They were members of the Engineering Products Group of the Society of Industrial Artists, and the purpose of the visit was to study the latest techniques in die casting. The firm's die casting foundries contain a large number of the most modern hot and cold chamber machines, including one of the largest in the country. There is also a new and fully equipped metallurgical laboratory. Members of the party discussed the design of components for suitability as die castings with some of the firm's technicians, and among the products they saw were wireless and television components, medical apparatus, components for electric hand tools, stage spotlight casings, scissor handles, carburettor parts, dental sprays and aerosol projectors. They also saw the manufacture of soda syphons and a new automatic cream whipper, and visited the wire braiding section where specialised work in connection with covering pressure hose and other types of wire weaving is done.

A Hundred Years of Steelmaking

Andrews Toledo Celebrate Centenary

Tapping the 10-ton electric arc furnace.

SOMETIME in 1856—the exact date is not known—J. H. Andrew founded the firm which is today known as Andrews Toledo, Ltd., and which has, for the past eighteen years, been a member of the Darwins Group. The original premises were in Malinda Street, Sheffield, although the precise location is again unknown. Ten years later, a move was made to the present site in Neepsend Lane, the name of the firm then being Jno Hy. Andrew and Company.

History

In the early years, quality steels were produced by the crucible process, and by 1870 there were in operation twelve crucible furnaces, with twelve holes to each furnace. In that year, the first Toledo forge was erected, to be followed a year later by bar mills and a wire rod mill. The decade from 1870 to 1880, saw the introduction of the open hearth process—with one acid furnace initially—and the erection of a spring shop and the Clifton forge.

During the first World War, a notable development took place when, in 1915, an electric arc furnace was installed. Today, arc furnace melting is widely used—even for the production of tonnage steels it is beginning to compete with open hearth melting—but forty years ago it was something of a novelty. A further wartime development was the installation of a new wire rod mill in 1917, and shortly after the war the present wire rod mill was laid down. Further landmarks in the Company's rolling plant developments were the erection in 1927 of a new 12 in. bar mill and the installation of a cogging mill in 1931.

During the past five years, comprehensive development and reconstruction have been effected throughout the Toledo steelworks. Additional plant has been installed, the most important of which is a 10-ton Efco Heroult arc furnace. Further developments are being considered in an endeavour to provide even higher quality steels.

In 1856 Jno. Hy. Andrew & Co., Ltd., was formed, with Lord Charles Beresford as chairman. The managing director was Mr. H. H. Andrew, son of the founder, who had been honoured two years previously by being elected Master Cutler. The next stage in the Company's development was the formation in 1929 of Andrews Toledo, Ltd. In 1938, Andrews Toledo amalgamated with Darwins, Ltd., thus becoming the first company to join what is today known as the Darwins Group.

The Company has always been concerned with the production of quality steels in the form of billet, bar and wire rod, but at certain periods in its history, other products, such as springs, hammers, picks and shovels were manufactured. Production of these ancillary products ceased in 1928, but not before they had played a big part, not only in the Company's activities, but also in the export drives of the latter part of the nineteenth century. Large quantities of mining steels were sent to Australia for the Gold Rush, and the gold and diamonds of South Africa purchased considerable quantities of mining steels. Steels were produced for the Balkan and Asia Minor oil fields, and extensive quantities of steel for various purposes were sold to Japan. High speed steels, tool and die steels, mining steels, springs, steels for the textile industry, guns and rifle barrels, bayonets and swords were all produced in ever-increasing quantities while the double shear steel forged under the Clifton Works' hammers was in great demand by Sheffield cutlery manufacturers. During the period 1905–1918, Toledo steels were in such great demand that the Company's forging, cogging and rolling facilities were much overloaded, which resulted in steel produced at Toledo being sent to outside firms for manipulation. By the outbreak of the first World War the brand of Toledo steels, together with the distinctive trade mark—the Hand and the Scimitar—was famous throughout the world.

Present Day Activities

Today, the 13½ acre island site, bounded on one side by Neepsend Lane and on the other by the River Don, houses electric and open hearth steel furnaces, which provide the raw material for the wire rod and bar mills. The output covers a wide range of carbon, alloy and special steels for use in the aircraft, automobile and engineering industries. They include hardened and

Charging an open hearth furnace.

tempered steels, case-hardening steels, nitriding steels, spring steels, valves steels, etc., and many of these are available in the black or bright surface condition. A speciality is the production of high quality carbon steels for the wire industry.

Steelmaking

Open Hearth Plant

There are three open hearth furnaces of 20–25 tons capacity, of which two are in operation at one time, whilst the third is under repair. All are acid-lined and fired by producer gas. Charging is effected by a Wellman charger, with a pan capacity of 23 cwt. Refining is accelerated by the use of oxygen lancing, oxygen being piped from an oxygen tank, via evaporators to the furnace.

All open hearth charges are cast by the uphill or bottom pouring method into 11½ or 21½ cwt. ingots, the latter being occasionally used for forging at another works of the Darwins Group. The casting bay is equipped with 7 ton and 15 ton E.O.T. cranes.

Arc Furnace

For the production of a wide range of special steels particularly those requiring a low sulphur and phosphorus content, there is a comparatively new 3,500 kVA. 10 ton Efco Heroult electric arc furnace fitted with Rotodyne control of the 12 in. graphite electrodes. The shell is 11 ft. 6 in. in diameter and the lining comprises a graphitised dolomite hearth, magnesite tube side walls and a silica brick roof. The roof, complete with electrode control gear is capable of lifting and slewing to enable the charge to be inserted in one load. This is effected by means of a charging basket suspended from a 20 ton E.O.T. crane.

As in the case of the open hearth plant, piped oxygen is available for lancing, and is used to eliminate carbon in high chromium charges. Both top and bottom pouring methods are used for casting the 11½ and 21½ cwt. ingots, and the furnace is served by a 20 ton E.O.T. crane with a ladle sling in a lifting beam.

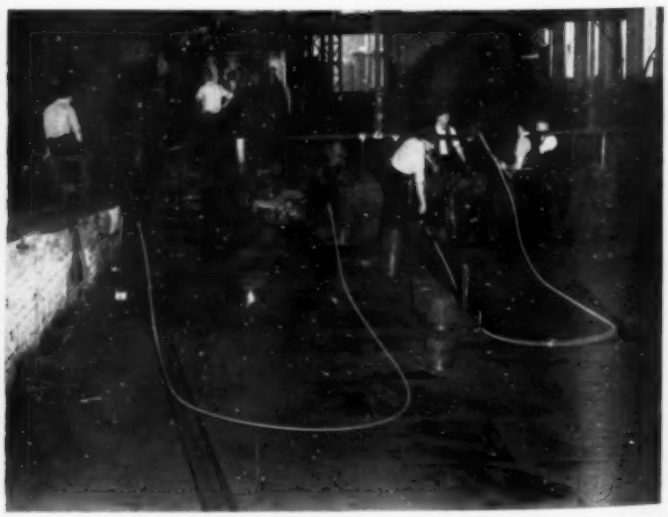
Laboratory.

A well-equipped chemical laboratory is available for the analytical control of the steelmaking process, and of the incoming raw materials.

Ingot Surface Treatment

Many of the special quality ingots require surface dressing before rolling. In most cases, this is carried out by a hot deseaming process in the deseaming department, which is equipped with a gas-fired ingot preheating chamber, a fume extraction unit, and ingot benches, and is served by a 3 ton E.O.T. crane. The hot deseaming process is suitable for the lower alloy steels and development work at present in progress is aimed at extending its range of usefulness. Steels which cannot at present be hot deseamed are planed or dressed in a square turning ingot lathe with a capacity of 11 in. × 4 ft. 6 in. long.

Three-high 25-in. cogging mill.



Wire rod mill with reheating furnaces in background

Catching and looping the wire rod for the next pass.

Handling the billet in the 14-in. bar mill.

Rolling

Cogging Mill

This is a three-high 25 in. unit fed by two continuous type ingot reheating furnaces fired by producer gas. The mill is driven by a 750 h.p. motor through reduction gearing which gives a final mill speed of 61·66 r.p.m. The billet sizes produced range from 3 in. to 6 in. square, and are cut to the required length by a 42 in. pendulum hot saw.

14 in. Bar Mill

This is the largest of the four bar mills and comprises four cross-country stands—three-high cogging, intermediate and oval stands, and a two-high finishing stand—driven by a 600 h.p. motor, through reduction gearing, at a speed of 108 r.p.m. The billets are reheated in a continuous type oil fired furnace and the bars are cut to lengths by a 36 in. pendulum hot saw, after which they are transferred to bogies via a bar cooling conveyor. The range of sizes rolled is $\frac{9}{16}$–$1\frac{3}{4}$ in. diameter, guide-rolled; $1\frac{3}{4}$–$4\frac{1}{2}$ in. diameter, hand rolled; and $\frac{9}{16}$–$1\frac{3}{4}$ in. across flats hexagons, guide rolled.

12 in. Bar Mill

The three three-high stands and two two-high stands of this mill are driven by a 600 h.p. motor, through reduction gearing, at a speed of 111 r.p.m. The billets are reheated in a continuous type oil fired furnace and the bars are cut to length by a 36 in. pendulum hot saw or by two sets of cropping shears. The mill can deal with rounds from $\frac{11}{16}$–$2\frac{1}{8}$ in. diameter; squares from $\frac{9}{16}$–$2\frac{1}{2}$ in.; flats from $1 \times \frac{1}{2}$ in. to $4\frac{1}{2} \times \frac{1}{2}$ in. (including spring steel); and various sections.

11½ in. Bar Mill

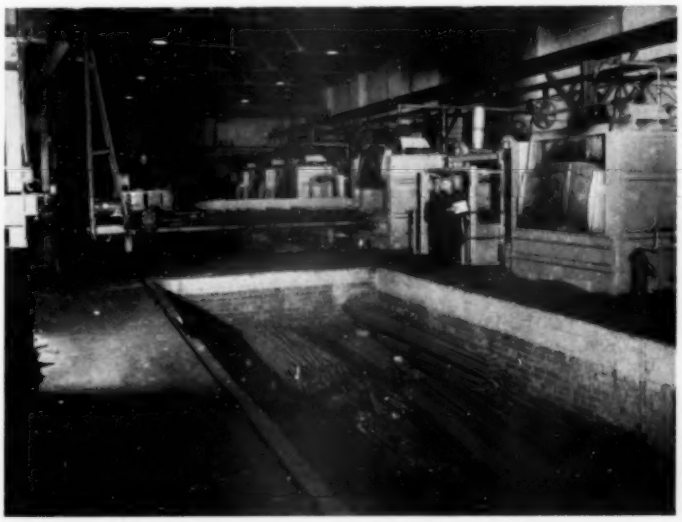
Used mainly for rolling special steels, this mill has two double-duo stands and two two-high stands, driven by a 350 h.p. motor, through reduction gearing, at a speed of 112 r.p.m. The billet reheating furnace is fired by a mechanical underfeed stoker, and the bars are cut by a 36 in. hot saw. The range of sizes rolled is $\frac{11}{16}$–$2\frac{1}{8}$ in. diameter; $\frac{3}{4}$–$1\frac{3}{4}$ in. square; $\frac{3}{4}$–$1\frac{1}{2}$ in. across flat octagons; $1 \times \frac{1}{2}$–2×1 in. flats; and various sections.

8½ in. Bar Mill

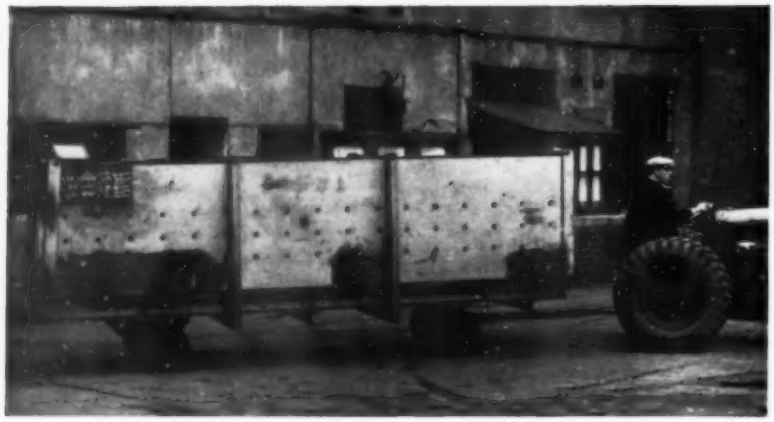
Double-duo stands are also a feature of this mill, there being two of them in addition to a two-high stand. The mill is driven by a 200 h.p. motor, through reduction gearing, at a speed of 210 r.p.m. The billets are heated in an oil fired furnace and the bars are cut to length by a portable cropping machine. Special quality steels may be slowly cooled in tubes preheated by the waste heat from the reheating furnace. The range of sizes produced in this mill is $\frac{1}{4}$–$\frac{5}{8}$ in. diameter; $\frac{1}{4}$–$\frac{1}{2}$ in. square; 0·250–0·590 in. across flats hexagons and octogons; $\frac{1}{2} \times \frac{1}{8}$ in.–$\frac{7}{8} \times \frac{3}{8}$ in. and $\frac{3}{8} \times \frac{7}{32}$–$1 \times \frac{1}{2}$ in. flats; and various sections.

Wire Rod Mill

Consisting of cogging, intermediate and finishing stands, the wire rod mill is fed by billets from one of two oil fired reheating furnaces. A 400 h.p. motor drives the three-high cogging stand, through a reduction gear, at a speed



General view of the heat treatment department.



Mobile furnace for transferring special steels from the mills to the heat treatment department.

of 125 r.p.m., and the intermediate stand, also three-high, runs at 206 r.p.m., driven by a 300 h.p. motor. From the intermediate stand, the rod passes to the eight two-high finishing stands, driven by a 650 h.p. motor at a speed of 366 r.p.m., and thence to the electrically driven coilers. Special wire rod is produced in a variety of qualities, including 0·15–1·40% carbon steels, free-cutting mild steel, a full range of En specifications, and nickel-iron alloys. The sizes range from 5 s.w.g.–$\frac{5}{8}$ in. diameter ; 6 s.w.g.–$\frac{17}{32}$ in. square ; $\frac{3}{8}$ in. × 12 s.w.g.–1 in. × 10 s.w.g. flats ; and hexagons and special sections by request.

Heat Treatment

For the heat treatment of bars prior to despatch from the works, there are eight modern gas fired furnaces, each having an automatic temperature controller and a temperature recorder. Two of these furnaces are used mainly for annealing of ingots and close annealing, the remaining six being available for normalising, hardening and tempering, etc. For the latter operation, the shop is provided with two oil quenching tanks and one water quenching tank in all of which are fitted means of continuously agitating the charge in the bath. The furnaces are charged by a 3 ton electrically driven ground charger, and are served by a 3 ton E.O.T. crane.

Mention should be made here of the mobile furnaces, or hot chambers, which are preheated by gas and used to transfer special steels from the mills to the heat treatment department.

Finishing

Adjacent to the heat treatment department is a warehouse which is fully equipped for finishing and inspection. The facilities include bar reeling machines, straightening machines, section straightening machines, cropping shears, cold sawing machines and a Hydrasurge cutting-off machine. The loading and unloading of materials is carried out with the aid of one 5 ton and two 3 ton E.O.T. cranes.

Inspection and Testing

Inspection of finished material is carried out in the warehouse, where the facilities include equipment for wet magnetic crack detection. There is also a test house to which samples can be forwarded to check that the mechanical properties are to specification. This department is also responsible for general investigation work in connection with the Company's processes and products, and for the control of quality. To this end, samples from production are subjected to sulphur printing, deep etching and microexamination. Facilities are also available for flaw detection by the magnetic and ultrasonic methods, which are used for both testing and development work.

Research Facilities

As a member of the Darwins Group, Andrews Toledo also has available the services of a research laboratory engaged in the development of new alloys and processes ; the inspection of raw materials and finished products ; the maintenance of works technical services and supervision of metallurgical processes ; and the provision of an advisory service for customers.

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80 Ton Arc Furnace for Scotland

Wm. Beardmore & Co., Ltd., steelmakers and engineers, Parkhead, Glasgow, have received approval from the Iron and Steel Board for a major reconstruction of their steel melting shop. The scheme, which will take about five years to complete, involves the replacement of the Company's open hearth furnaces by electric arc furnaces at an estimated cost of £1,600,000, and will be financed from the Company's own resources.

This scheme will facilitate the production of the higher qualities of steel now being called for, and, when completed, will increase the steelmaking capacity of Parkhead melting shop by 30%. In order to maintain output during reconstruction, the work will be carried out in stages, the first will be the installation of an Efco 80-ton electric furnace. This furnace—which is of course the key unit—is being built and supplied by the Electric Furnace Co., Ltd., of Weybridge, and will be larger than any furnace operating in this country today. It will have a productive capacity of at least 70,000 tons of carbon and alloy steels per year, and will have a transformer rating of 18 MVA, while the furnace itself will possess special design features to give increased output and more accurate control.

The Electric Furnace Co., Ltd., pioneers of the first arc furnace installations in this country some forty years ago, are at present building a 60-ton unit for the South African Iron and Steel Corporation in addition to this 80-ton furnace for William Beardmore.

The Effect of Composition on the Mechanical Properties of Al-Si-Cu-Mg Casting Alloys

By M. G. Gittins* and W. E. Mew†

(On Behalf of the Technical Committee of ALAR)

During recent years there has been some change in wrought aluminium production from copper-bearing alloys to copper-free alloys usually containing a somewhat higher magnesium content. This change has had repercussions in the manufacture of secondary alloys, and this paper describes a series of tests on cast aluminium-silicon-copper-magnesium alloys in which the influence of copper and magnesium content on the mechanical properties has been examined.

THE alloy to B.S.1490 LM 4 is by a considerable amount the most widely used aluminium casting alloy in the U.K. It is generally produced from scrap aluminium emanating from the fabrication of wrought alloys, such as sheet cuttings, section scrap, and borings and turnings from bars and forgings. During recent years, there has been some change in the wrought aluminium production from copper-bearing alloys to copper-free alloys usually containing somewhat higher magnesium contents. This change has rendered the production of LM 4 alloy from the scrap arising increasingly expensive, as more magnesium has to be removed and copper additions have to be made to arrive at the specified composition. In order to make the best use of the scrap arising, it seems desirable to contemplate adjustments in the composition of the LM 4 alloy, if these can be made without impairing the properties of the material. It is with the intention of providing basic information for this case that the experiments described in this paper were undertaken.

An effort was also made to find a more reproducible and easily measured method of testing the ductility of cast specimens of alloys with low elongation. This is dealt with in the appendix at the end of the paper.

Summary of Published Work

As the first step in the investigation, a survey was made of published information on alloys within a similar range of compositions, and it was found that a number of workers had examined the properties of cast alloys containing approximately 4–6% silicon, with up to 4% copper and varying amounts of magnesium, manganese, iron and other impurity elements.

Dix and Lyon,[1] as early as 1922, tested the mechanical properties of aluminium-silicon-copper alloys, but their alloys contained no magnesium. Archer and Kempf[2] in 1930, however, determined the tensile properties of a series of alloys, in the as cast and solution heat treated conditions, containing 5% silicon, 0–1·25% copper 0·1–0·9% iron, and 0·35% magnesium. Later, Quadt[3] investigated the effect of small amounts of nickel and zinc on the alloy containing 1% copper, and also demonstrated the beneficial effect of manganese when the iron content was high; this work was carried out on as cast and solution treated specimens. Bollenrath and Groeber[4] also studied the properties of cast aluminium-silicon-copper alloys in the solution heat treated condition; two series of alloys were mechanically tested, each with 4–7% silicon plus copper, one made from pure metals and the other from secondary raw materials. Siebel[5] examined the composition range: 4·5% silicon, 0·5–2% copper, 0–0·6% magnesium, with controlled additions of manganese and iron. He determined the tensile properties and hardness of sand cast specimens in the as cast and solution treated conditions. The effects o manganese, iron and zinc on the characteristics of an aluminium, 5% silicon, 1% copper, 0·5% magnesium alloy were investigated by Achenbach,[6] but mechanical properties only after solution heat treatment are reported, and the paper is chiefly concerned with the casting characteristics of the alloys.

The mechanical properties of alloys containing 6% silicon, 0·5% magnesium with nil and 1% copper are the subject of an unpublished report of International Alloys, Ltd.[7] In this programme, specimens were tested in the as cast condition at varying times up to 28 days after casting; others were tested in the precipitation and fully heat treated conditions. At the same time the effect of the rate of cooling after casting of test bars was investigated. More recently Scheuer, Williams and Wood[8] made a detailed survey of the mechanical properties of sand cast specimens of alloys containing 2–4% copper, 2–6% silicon and 0·25% magnesium, together with the amounts of other elements usually present in alloys such as LM 4. The tests were made solely on alloys in the as cast condition. The age hardening at room temperature of aluminium-silicon-copper alloys has been examined by Wood[9] and by Smith.[10]

Finally, much is known from practical experience of alloys of the type 5% silicon, 0·5% magnesium with nil and 1% copper, since they are the subject of British standards, being, respectively, LM 8 and LM 16 in B.S.1490, and are widely used in the U.S.A. as Alloys 356 and 355. In the revised edition of B.S.1490, published in November, 1955, an as cast condition for LM 8 has been added, but, apart from this exception, both alloys are specified in the U.K. and U.S.A. only in heat treated conditions. Foundry experience has

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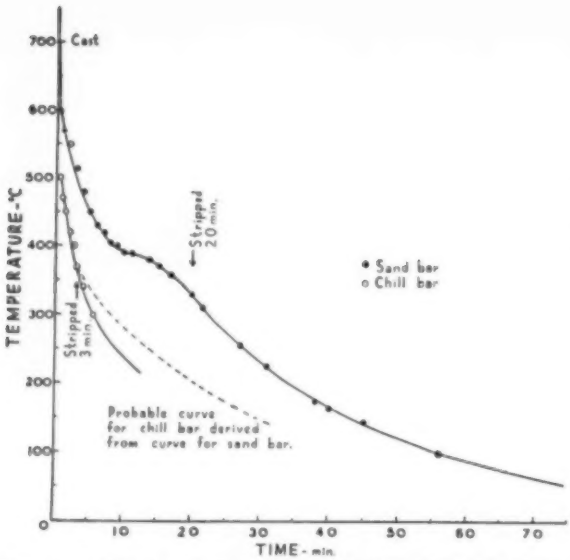

Fig. 1.—Cooling curves of sand and chill cast test bars.

demonstrated the very good casting characteristics of both alloys.

An attempt to summarise the results obtained from the above sources revealed the wide diversity of conditions under which the tests have been conducted. Most of the workers have been concerned with fields of composition which include alloys containing 5–6% silicon, 0–1% copper and up to 0·5% magnesium, but some of the alloys, e.g., those examined by Scheuer, Quadt and Achenbach, contained impurity elements in the normally encountered amounts, whilst others, e.g., Siebel and Archer and Kempf, worked with purer alloys. Bollenrath and Groeber tested both types of alloy and found superior strength and hardness in the normal alloy but greater ductility in the purer alloy.

Comparison of mechanical properties is made particularly difficult by three factors :—

(1) The sand cast test bars used varied in size from the extremes of the American 0·5 in. diameter cast-to-shape specimen to the 1 in. diameter cylindrical bar used by British investigators ; for chill castings very little information is available on shape and mass of moulds.

(2) The tests made by Wood and others show that the aluminium-silicon-copper alloys age harden at room temperature after casting, and that the tensile strength may increase and the elongation fall significantly—to a greater degree if magnesium is present—during the first few days after casting. In the work reviewed above, mechanical tests were carried out within a few hours of casting only by Scheuer and his colleagues. Archer and Kempf allowed all their specimens to age for four days ; Bollenrath did not control the time between casting and testing, but states that it was not less than five days, and Siebel's tests were made several weeks after casting ; mechanical properties plotted by Quadt relate to tests made one to three days after casting.

(3) The report of International Alloys, Ltd., shows that the rate of cooling in the mould of both sand and chill cast bars can affect the subsequent rate of age hardening and response to precipitation heat treatment. There is no evidence that any of the investigators other than Scheuer, Wood and Smith accurately standardised the cooling conditions for their test bars.

The mechanical properties of specimens in the fully heat treated conditions—to which factors (2) and (3) above would not apply—can be correlated on a common basis, if an allowance is made for test bar sizes, and they have been sufficiently examined to make any further work unnecessary at the present time.

The mechanical properties of as cast specimens on the other hand have been much less studied, and the results cannot easily be compared for the reasons already given. None of the investigators has examined the effect of age hardening or precipitation treatment on the alloys within the chief field of interest, i.e., with up to 2% copper. Since the alloy LM 4 is used only in the as cast condition (except the special version, LM 22, which is solution heat treated) this is the condition for which mechanical properties are most required for the range of composition to be explored, and, since the alloys are known to age harden, the effect of this change on the mechanical properties must also be measured. Finally, most of the work has been carried out on sand cast bars, but for an alloy of this type the properties of chill castings are equally important.

On the basis of this review, it was decided to undertake the series of tests outlined below, which would enable the mechanical properties of alloys over a wide range of composition to be compared on a standardised basis.

Programme of Tests

The composition of LM 4 castings is

Cu %	Mg %	Si %	Fe %	Mn %	Ni %
2·0–4·0	0·15 max.	4·0–6·0	0·8 max.	0·3–0·7	0·3 max.

Zn %	Pb %	Sn %	Ti %	Al %
0·5 max.	0·1 max.	0·05 max.	0·2 max.	Remainder

It will be noted that the specification allows fairly wide limits for impurities, and it was thought advisable to eliminate some of the variables in this respect. An examination was therefore made of the effect of varying copper and magnesium additions, both separately and together, to a 5–6% silicon alloy made from primary aluminium of 99·5% purity. It was thought that the silicon content should remain unchanged throughout the series, as this constituent is not in practice influenced by the type of scrap available. Furthermore, it was not to be expected that any benefit would be derived from altering this percentage. The compositions tested were as follows :—

Silicon	5·8%
Iron	0·3%
Copper	0%, 1·0%, 2·0% and 4·0%
Magnesium	0%, 0·15%, 0·35% and 0·60%

An additional composition containing 0·5% copper and 0·35% magnesium was also tested.

The difference in the scale of copper and magnesium additions should be noted. Previous work has shown that the amounts of magnesium required to change the mechanical properties are much less than those of copper.

Sand and chill cast test bars were subjected to tensile, elongation and bend tests in the as cast condition, that is, within four hours after casting ; after four weeks

TABLE I.—EFFECT OF COMPOSITION ON MECHANICAL PROPERTIES OF SAND CAST BARS.

		0% Magnesium				0·15% Magnesium				0·35% Magnesium					0·60% Magnesium			
	Copper %	Nil	1·0	2·0	4·0	Nil	1·0	2·0	4·0	Nil	0·5	1·0	2·0	4·0	Nil	1·0	2·0	4·0
Tensile Strength (tons/sq. in.)	As cast	8·0	9·0	9·3	9·9	8·1	8·9	9·5	9·9	8·2	9·1	9·2	9·6	10·1	8·4	8·7	9·1	9·3
	Aged 4 weeks	7·6	8·6	9·3	10·1	8·2	9·9	10·7	11·6	8·6	9·8	10·3	10·8	11·4	8·8	9·4	9·8	9·5
	Heat treated	7·7	9·7	9·6	10·5	10·5	12·1	12·7	13·9	11·5	13·2	13·5	13·5	14·3	12·6	12·5	12·8	11·5
Elongation (%)	As cast	6·8	4·9	4·0	2·2	5·6	3·7	2·5	1·8	3·0	3·3	3·1	2·0	1·6	1·9	1·8	1·6	0·8
	Aged 4 weeks	5·4	4·0	3·5	2·2	4·5	2·7	2·1	1·3	2·3	2·4	1·9	1·3	0·7	1·4	1·3	0·7	0·6
	Heat treated	5·7	4·2	3·5	2·1	1·5	1·0	0·8	0·4	0·6	0·4	0·6	0·6	0·3	0·3	0·4	0·3	0·2

TABLE II.—EFFECT OF COMPOSITION ON MECHANICAL PROPERTIES OF CHILL CAST BARS.

		0% Magnesium				0·15% Magnesium				0·35% Magnesium					0·60% Magnesium			
	Copper %	Nil	1·0	2·0	4·0	Nil	1·0	2·0	4·0	Nil	0·5	1·0	2·0	4·0	Nil	1·0	2·0	4·0
Tensile Strength (tons/sq. in.)	As cast	9·4	11·7	12·7	13·8	10·5	12·2	13·1	13·0	11·3	11·8	13·1	13·5	13·7	10·6	13·0	12·4	14·2
	Aged 4 weeks	9·6	11·3	12·8	14·2	11·2	13·1	14·3	14·7	12·0	11·6	14·2	14·6	15·2	12·1	12·9	13·4	15·9
	Heat treated	9·3	11·3	12·5	14·4	12·8	15·1	15·2	16·7	15·0	15·6	16·7	16·8	17·5	14·6	15·3	16·6	17·2
Elongation (%)	As cast	13·2	13·0	8·4	6·5	19·2	9·1	6·6	4·3	8·8	6·4	8·1	5·4	4·6	4·3	5·5	4·6	2·8
	Aged 4 weeks	16·8	10·4	8·9	6·1	17·3	6·1	5·1	2·7	7·4	4·1	6·0	5·1	2·5	6·6	3·5	2·3	2·7
	Heat treated	13·8	9·8	8·6	5·0	9·6	3·1	2·3	1·6	2·9	1·8	2·5	1·7	1·1	2·3	1·2	1·4	1·3

natural ageing, and after precipitation heat treatment (16 hours at 160° C.).

Melting and Casting Procedure for Test Specimens

Four basic alloys, having the compositions given below, were prepared in a half-ton capacity melting crucible.

	Cu %	Mg %	Si %	Fe %
Basic Alloy 1	3·93	0·60	5·71	0·31
Basic Alloy 2	3·93	—	5·99	0·30
Basic Alloy 3	0·03	—	5·75	0·31
Basic Alloy 4	0·03	0·57	5·74	0·24

The final alloys were prepared by re-melting and blending portions from these basic alloys to give the required composition. The analyses of two of these, which are given to indicate the general level of impurities and the accuracy of the blending are shown below.

Alloy	Cu %	Mg %	Si %	Fe %	Mn %
0·5 Cu/0·35 Mg..	0·54	0·35	5·80	0·28	<0·01
	Ni%	Zn %	Pb %	Sn %	Ti %
	<0·01	0·01	<0·01	<0·01	<0·01
	Cu%	Mg %	Si %	Fe %	Mn %
2 Cu/0·15 Mg..	2·03	0·15	5·71	0·26	<0·01
	Ni %	Zn %	Pb %	Sn %	Ti %
	<0·01	0·01	<0·01	<0·01	<0·01

The re-melting was carried out in a small bale-out crucible, and before pouring the test bars the metal was degassed with nitrogen at a temperature of 750° C., and allowed to cool to 720° C., at which temperature the metal was removed with a pre-heated hand ladle. Standard sand cast bars, as specified in B.S. L101, were poured at 690° C., the sand moulds being tilted to 30° from the vertical whilst casting and returned to the perpendicular when filling the head. A uniform rate of pouring was maintained, the parallel portion of the bar taking between five and ten seconds to fill. Chill cast bars were poured at 710° C., into the horizontal wedge-shaped moulds specified in B.S. L 101. The moulds were pre-heated to 100° C. by immersion in boiling water, the temperature being adjusted to this figure prior to the pouring of each bar.

As there is a strong possibility of mechanical properties of test bars in this type of alloy being dependent on the rate of cooling after solidification, owing to precipitation processes, the time between casting and stripping test bars was standardised in accordance with conditions in normal practice with sand castings and die castings of medium size. For sand cast test bars a stripping time of 20 minutes was selected, and for chill cast bars a stripping time of three minutes. After a further 20 minutes in still air, the test bars were quenched in water.

Actual measurements of the temperature drop in the central part of the test bars gave the curves shown in Fig. 1. The sand cast bar was stripped after 20 minutes and the cooling in still air followed for a further 20 minutes. In the case of chill casting, the bar remained in the mould. As the temperature at stripping was near to that of the sand cast bar, the air cooling curve of this latter one applies also to the chill cast bar, and this is indicated in the figure.

It can be seen from Fig 1 that the sand cast bar is stripped at about 330° C. and the chill cast bar at about 370° C. In the case of the sand cast bar, a cooling delay occurs in the region of 400° C., which seems to indicate precipitation processes. In the chill cast bar, no indications of this delay are visible.

At the moment of stripping, there seems to be no pronounced change in the rate of cooling in the case of sand cast bar. In the chill cast bar, the rate of cooling is considerably slower after stripping than immediately before.

The final quenching in water, 20 minutes after stripping, occurs at about 150° C., with both the sand cast and chill cast bars.

Presentation of Results

Tables I and II.—These are summary tables of all results. The figures given are the average of three or four test bars in each case. Test bars which showed any evidence of inclusions or other faults were discarded.

TABLE III.—CHANGES IN MECHANICAL PROPERTIES UPON PRECIPITATION HEAT TREATMENT.

		Tensile Strength (expressed as percentage of "as cast" value)		Elongation (expressed as percentage of "as cast" value)	
Cu (%)	Mg (%)	Sand	Chill	Sand	Chill
Nil	Nil	− 4	− 1	− 16	+ 4½
	0·15	+ 50	+ 22	− 73	− 50
	0·35	+ 40	+ 33	− 80	− 67
	0·60	+ 50	+ 38	− 84	− 47
1·0	Nil	+ 1	− 3½	− 14	− 25
	0·15	+ 36	+ 24	− 73	− 66
	0·35	+ 47	+ 28	− 81	− 69
	0·60	+ 44	+ 18	− 76	− 78
2·0	Nil	+ 3	− 1½	− 13	+ 2½
	0·15	+ 34	+ 16	− 68	− 65
	0·35	+ 41	+ 24	− 70	− 69
	0·60	+ 41	+ 34	− 81	− 70
4·0	Nil	+ 6	+ 4½	− 4½	− 23
	0·15	+ 40	+ 28	− 78	− 63
	0·35	+ 41	+ 28	− 81	− 76
	0·60	+ 24	+ 21	− 75	− 54

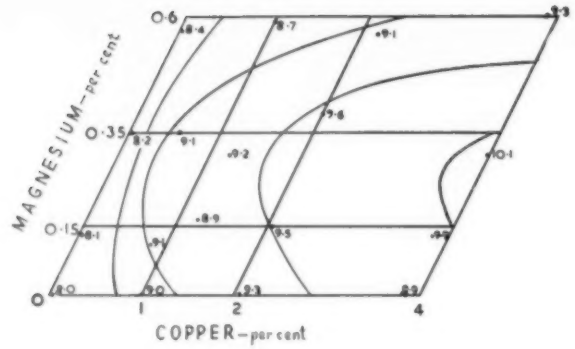

Fig. 2.

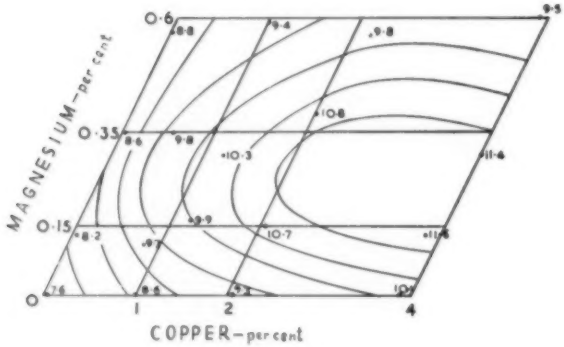

Fig. 3.

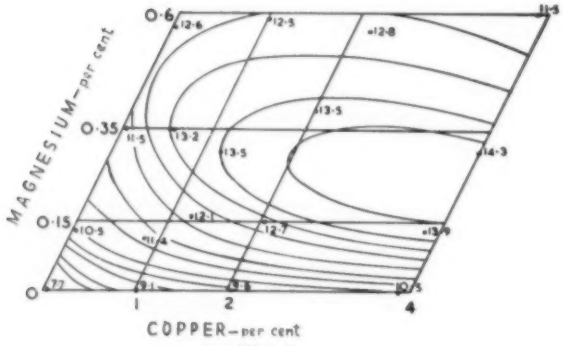

Fig. 4.

Figs. 2–4.—Effect of composition on the tensile strength of sand cast test bars. Contours at intervals of 0·5 ton/sq. in., ranging from 8·5 to 10 tons/sq. in. in Fig. 2 (as cast); from 8 to 11 tons/sq. in. in Fig. 3 (aged 4 weeks); and from 8·5 to 14 tons/sq. in. in Fig. 4 (heat treated).

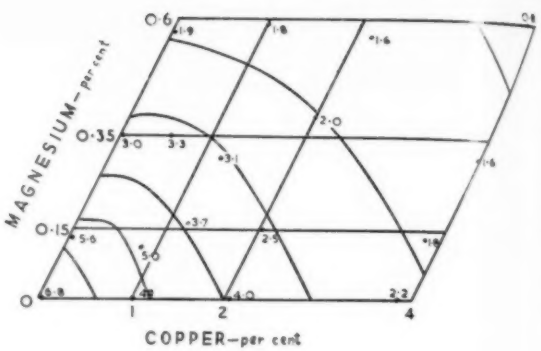

Fig. 5.

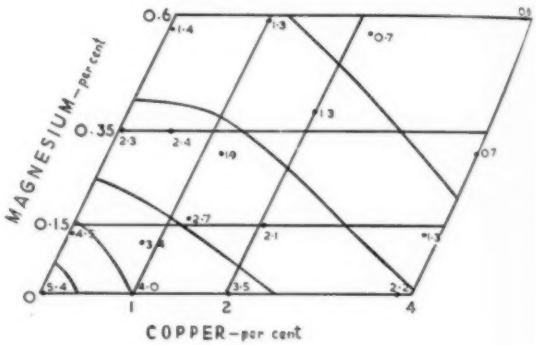

Fig. 6.

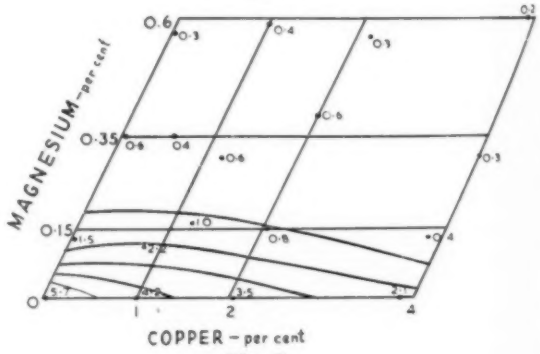

Fig. 7.

Figs. 5–7.—Effect of composition on the elongation of sand cast test bars. Contours at intervals of 1%, ranging from 1 to 6% in Fig. 5 (as cast); and from 1 to 5% in Fig. 6 (aged 4 weeks); and Fig. 7 (heat treated).

It was found, as expected, that, when testing large numbers of cast test bars, there was some scatter in the results, and with this in mind each set of figures has been carefully examined and some repeat tests made where the agreement between similar bars was not thought good enough.

Table III.—This table compares the changes in properties of the various alloys resulting from precipitation treatment.

Figs. 2–13.—These are contour graphs showing the effect of increasing copper and magnesium on the tensile strength and elongation in the as cast, naturally aged and precipitation treated conditions, both for sand cast and chill cast bars. Attention should be drawn to the different scales used for copper and magnesium as mentioned under heading " Programme of Tests."

Fig. 14.—This shows the contour graphs from Figs. 2 and 5 superimposed, with the shaded area giving the limits of composition within which a tensile strength of 9 tons/sq. in. and an elongation of 2% can be obtained in the as cast condition from sand cast test bars.

Fig. 15.—This is a graph corresponding to Fig. 14, but based on values obtained after four weeks natural ageing.

These contour graphs have been drawn as nearly in line as possible with the actual plotted figures obtained in the tests.

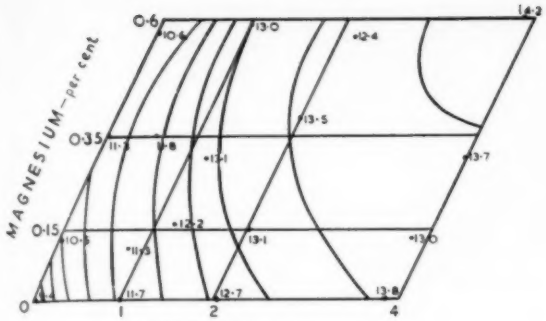

Fig. 8.

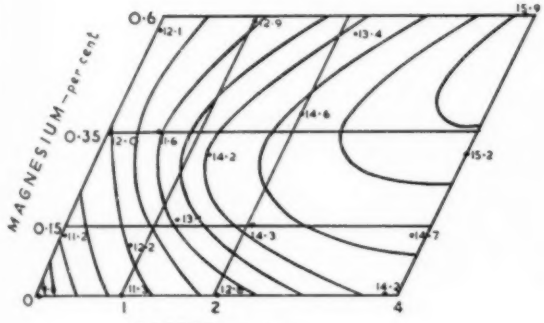

Fig. 9.

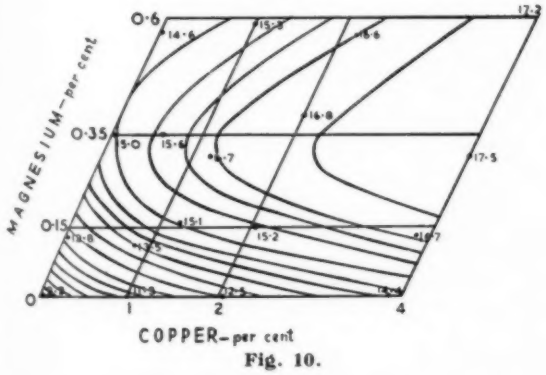

Fig. 10.

Figs. 8–10.—Effect of composition on the tensile strength of chill cast test bars. Contours at intervals of 0·5 tons/sq. in., ranging from 10 to 14 tons/sq. in. in Fig. 8 (as cast); from 10 to 15·5 tons/sq. in. in Fig. 9 (aged 4 weeks); and from 9·5 to 17 tons/sq. in. in Fig. 10 (heat treated).

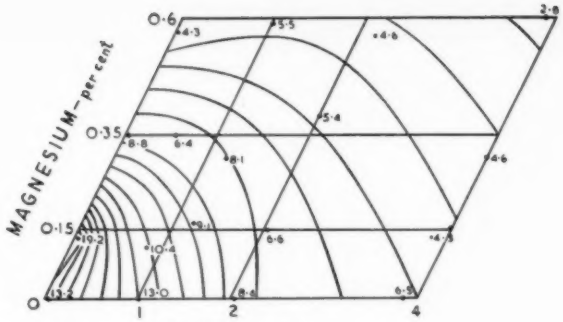

Fig. 11.

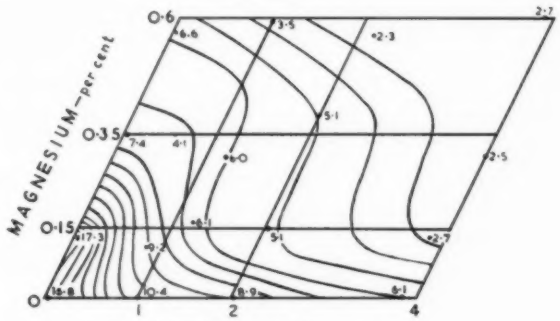

Fig. 12.

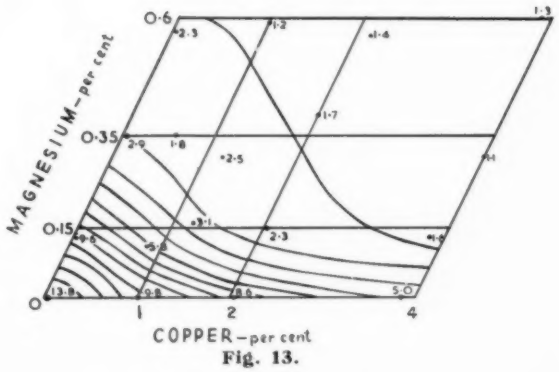

Fig. 13.

Figs. 11–13.—Effect of composition on the elongation of chill cast test bars. Contours at intervals of 1%, ranging from 3 to 19% in Fig. 11 (as cast); from 3 to 17% in Fig. 12 (aged 4 weeks); and from 2 to 13% in Fig. 13 (heat treated).

Examination of Results

(*a*) There is a definite deterioration in the tensile strength in both naturally and artificially aged specimens, as the magnesium content exceeds about 0·3% in the alloys with copper contents above 1%.

(*b*) The elongation falls regularly with increasing additions in all cases, whereas for maximum tensile strength there is an optimum mangesium content which depends to some extent on the copper content. This is especially pronounced in the naturally and artificially aged specimens.

(*c*) Natural ageing affects to the greatest degree the elongation of alloys in the range of above 1% copper and between 0·1% and 0·35% magnesium. Artificial ageing reduces particularly the elongation of the alloys with the lower copper contents, which have initially a high elongation.

(*d*) The addition of the first 0·15% of magnesium has more effect than subsequent additions, especially in the chill cast bars. Copper has also more effect in the lower range, but is still effective in the region of about 4%.

(*e*) The influence of hardening elements is generally greater in chill cast bars than in sand cast bars, e.g.,

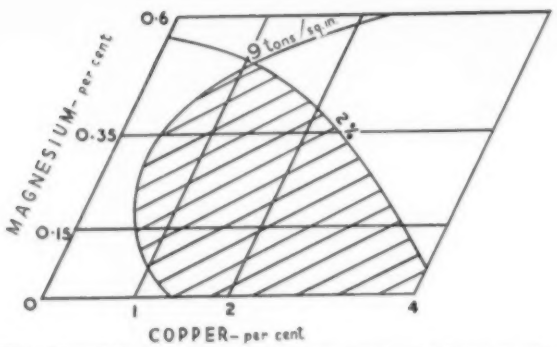

Fig. 14.—Effect of composition on mechanical properties of sand cast test bars, determined 4 hours after casting. The shaded area represents the composition range in which minimum values of 9 tons/sq. in. and 2% can be attained for tensile strength and elongation, respectively.

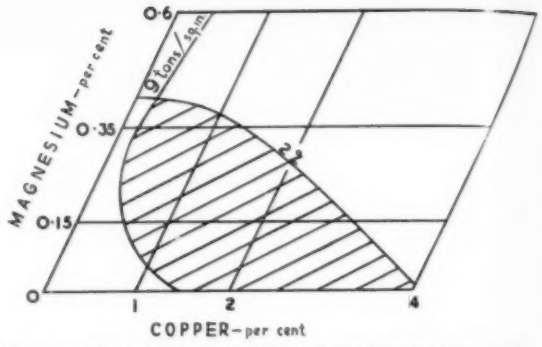

Fig. 15.—Effect of composition on mechanical properties of sand cast test bars, determined 4 weeks after casting. The shaded area represents the composition range in which minimum values of 9 tons/sq. in. and 2% can be attained for tensile strength and elongation, respectively.

Element	Increase in Content %	Increase in Tensile Strength (tons/sq. in.)	
		Sand Cast	Chill Cast
Magnesium	0 to 0·35	0·5	2
Copper	0 to 4·0	1–2	2·5–4

A similar difference can be seen between sand and chill bars in the naturally aged condition.

(*f*) The change in properties produced by precipitation heat treatment is greater in sand cast bars than in chill cast bars. This is illustrated in Table III.

Conclusions

(1) It would appear that if the copper content of the LM 4 alloy were to be reduced to around 1%, a range of composition could still be found which would give mechanical properties within the LM 4 specification.

(2) The practical top limit for magnesium when both copper and silicon are present is approximately 0·3%, if mechanical properties similar to those of LM 4 are desired without heat treatment. (It was also noted that where the magnesium was over 0·3% there was a tendency towards inclusions, probably of oxide films, in the test bars. This is usual in alloys containing magnesium). If the magnesium content is very low, with a low copper content also, then the alloy becomes much more ductile and softer than is normal for LM 4.

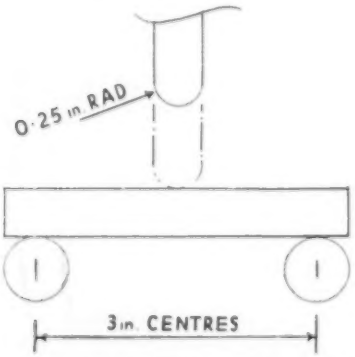

Fig. 16.—Sketch showing method of carrying out bend test.

(3) Comparing the results obtained with data available for similar but less pure alloys shows that the higher purity alloys have better elongation, but that the tensile strengths are little different.

(4) Age hardening is a normal characteristic of all these alloys, except where copper and magnesium contents are very low. The four weeks natural ageing period has been taken as the time required for practical completion of ageing. This assumption has been based on previous work on the natural ageing of these alloys. (It is not strictly true where magnesium is entirely absent). The indications are that with the pure alloys the tensile strength will increase by about 0·5 tons/sq. in., and the elongation will fall by 0·5% (after the natural ageing period.) From Fig. 15 it can be seen that the effect of natural ageing is to reduce the effective range of copper and magnesium within which the specified mechanical properties of LM 4 can be obtained when considering castings in practical use.

Appendix—Note on Bend Test

L 101 type sand and chill cast bars were each machined to $\frac{1}{2}$ in. diameter and parted into two $3\frac{1}{2}$ in. lengths. These specimens were supported on anvils and bent in the manner shown in the sketch attached (Fig. 16). The second specimen was turned about its central axis through 180° in order to average out directionality. The angle of bend on average was of the order of 3° for each 1% of elongation, and was directly proportional to the elongation. The degree of scatter between specimens from the same bar was similar to the scatter on the elongation figures. It was not found that the angle of bend was much more readily measured than the extension of the standard test pieces, although an improvement might be expected with more refined equipment. Bend tests were carried out on every specimen, but it was felt that this test did not show any marked advantage over the normal elongation tests and the results have not been included.

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Production of Special Alloys

New Telcon Works at Crawley

SIX years ago, The Telegraph Construction and Maintenance Co., Ltd., published, with the title of "The Telcon Story," a most interesting book recording the history of the company during its first hundred years. Perhaps the most important feature of that history, from a metallurgical point of view, was the setting up in the 1920s of a Metals Department to cater for the Company's needs for special magnetic materials for submarine communication cables. At that time the Company was engaged on the manufacture of telegraph cables connecting the island of Cocos with the Australian mainland at Perth, and, subsequently, for the Vancouver to Fanning Island link. It had been shown many years previously that, by winding a wire or tape of easily magnetisable material round the copper conductor of such a cable, much more traffic could be handled, and in 1921–22 a group of technologists joined the Company specifically for the purpose of developing suitable magnetic materials. Included in this group were the present Managing Director of what is now the Metals Division, Mr. W. F. Randall, and the Manager of the new Crawley factory, Dr. G. A. V. Sowter.

Early Developments

As a result of investigations, there was evolved at that time a material which has become a household word in the electrical engineering and communications industry —Mumetal. It is a striking tribute to the effectiveness of the early experimental work that this alloy is in ever increasing demand even to-day, the production being limited only by the availability of the major constituent —nickel. Mumetal actually consists of 76% nickel, 4% molybdenum, 12% iron and a small balance of other essential elements. Other nickel alloys for magnetic applications evolved in the 1920–30 era are still widely utilised with the familiar names of Radiometal and Rhometal. During this period also, the Metals Department began the production of nickel-chromium and nickel-chromium-iron resistance alloys for electrical heating elements, and here too, present day demand exceeds the availability of supplies.

In the early 1930s, the merits of the nickel-iron series of high permeability magnetic alloys began to be recognised by the electrical engineering industry, and Mumetal was widely adopted for instrument transformers for the measurement of electrical power in distribution systems. This practice has been widely adopted in the United Kingdom, throughout the Commonwealth, and in many countries of the world, and to-day Mumetal cores are in ever increasing demand. They are mainly embodied in switchgear of all types, and find a not insignificant place in modern atomic energy power stations.

In the few years immediately preceding the war, the Metals Division concentrated on magnetic and resistance alloys, but increased its team of technicians with the object of extending the range of its products. Strengthened by the arrival of the present Technical Manager, Dr. H. H. Scholefield, in 1941, there have been produced a number of Telcon metals which are playing an important part in the export drive, automation, electronic computing, lightweight aircraft generators and temperature control devices.

Need for Expansion

During the war, the metals production facilities at Greenwich were severely taxed because all the alloys had found wide use in armaments of all types. As a result of the experience of working 24 hours a day, 7 days a week, for several years, there arose the idea of building a new factory where production could be carried out on the most up-to-date plant available and where working conditions—and living conditions—would be considerably improved. This idea has now been put into practice in a new works at Crawley, in Sussex, which is now in full production.

Crawley was chosen as the site for one of the new towns to be set up to deal with the overspill population problem, and there are now more than 30,000 inhabitants as compared with a pre-war figure of about 7,000. A factory estate has been established on the outskirts, and it is there that the Metals Division of The Telegraph Construction and Maintenance Company has built the new Telcon Works, which are situated on a site of about 10 acres. The foundation stone was laid by the late Lord Colgrain, Chairman of the Company, on June 22nd, 1953, and the first wheels turned on October 16th, 1954.

The New Factory

An area of 106,000 sq. ft. is devoted to factory space and 20,000 sq. ft. to offices and laboratories for research and development. The factory itself is rectangular in shape, 440 ft. long × 240 ft. wide, and consists of two central bays each 80 ft. wide and two side bays each 40 ft. wide. The whole central area is covered by a reinforced barrel roof with lights, and continuous windows at a high level above the roofs of the side bays run down both sides of the factory.

The factory has ample natural lighting and ventilation, and operation at night is made as efficient as by day by the effective artificial lighting using tungsten filament lamps in convector type reflectors. Heating is by well equipped oil-burning boilers situated in a modern boiler house on the east side. Apart from space heating, steam is also used for domestic hot water and for production purposes, the main process being the acid pickling plant located in one of the bays on the west side.

A 440 v. 3-phase power system is obtained by transforming an incoming 11 kV. supply in two substations situated in diagonally opposite sites on the north-east and south-west corners of the factory. There are several hundred motors installed in various parts of the plant, whose power ranges from ¼ to 500 h.p.; and for melting the alloys there are a number of high frequency motor generator sets.

Normally about 1½ million gallons of water would be required per week for cooling and pickling processes, but considerable economy has been effected by utilising a closed circuit system embracing an artificial pond which forms an artistic feature of the foreground of the factory. Since water costs 3s. 10d. per thousand gallons in

Teeming special alloy ingots.

Crawley, the saving in production costs, due to the pond, is appreciable. Water is taken from the pond and pumped into a large steel pressure vessel under air at 50 lb./sq. in. This provides a nearly constant head to the factory and, when the pressure drops to 42 lb./sq. in., the h.p. pumps start up and continue filling the vessel until the pressure is restored to 50 lb./sq. in. After use in the factory plant, the warm water is passed by gravity to an underground sump where it returns to the pond by a low pressure pump.

As detailed later, several furnaces in the factory are gas heated, and suitable mains have had to be installed to deal with a load of nearly 2 million cu. ft./week. A small building adjacent to the boiler house is used for the production of cracked ammonia for the furnace annealing atmospheres, and this is distributed by pipes under the road to the various plants involved. Compressors are situated within the factory for the supply of compressed air, which is used for a blacksmith's hammer, the making of moulds, shot blasting and various incidental processes.

In general, the siting of the plant within the factory has followed a definite plan, with materials entering near the south-east corner and leaving near the gate on the north-west side. The constituents of the various alloys are generally purchased as virgin metals, such as nickel, copper, iron, chromium, manganese, molybdenum, cobalt, silicon, aluminium, calcium, etc., although the beryllium used in the production of beryllium-copper is obtained as a 4% beryllium-copper master alloy. Minor constituents such as vanadium and titanium are purchased as ferro-alloys.

Melting

The main melting plant consists of 5 and 10 cwt. Efco induction furnaces supplied with power by motor generator sets. There is also a 5 cwt. vacuum melting and casting unit of the H.F. type, capable of operating at a pressure of 0·1–1 mm. Hg. This is mainly used for the production of super quality materials. Beryllium-copper is melted in a gas-fired tilting crucible furnace at the same end of the factory, but before the end of the year this operation will have been transferred to a separate building.

The metal from the induction furnaces is transferred to ladles for teeming into the ingot moulds. Bottom teeming is used, as in the steel industry, the ladles having a nozzle in the bottom and a refractory stopper by which flow is controlled. Strip production is mainly based on 9 in. square ingots weighing 340 lb. each.

Hot Strip Rolling

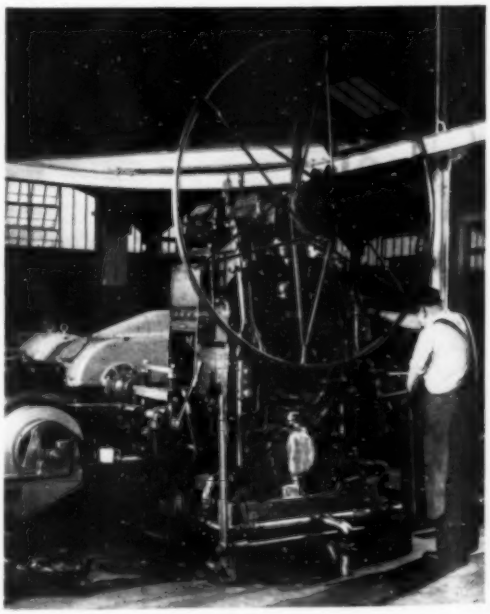
Ingots, whether destined for strip or wire, are heated in an Incandescent pusher-type furnace, prior to rolling in the Robertson three-high cogging mill, which is equipped for rolling either 6 in. × 2 in. slabs for strip, or $1\frac{3}{4}$ in. squares for eventual conversion to rod or wire. The slabs then pass, via a Thermic Revergen reheating furnace to a Robertson three-high hot strip mill, followed by a flattening mill.

The product from the hot strip mill requires further treatment before cold rolling can be attempted. The long strips are passed through a continuous sodium hydride descaling bath, and then scratch-brushed to remove scale and surface imperfections. They are then transferred laterally to the conveyor of a Metalectric continuous annealing furnace supplied with a cracked-ammonia atmosphere.

Cold Rolling

After annealing, the strip is ready for breaking down by cold rolling into thinner gauges. This is achieved on a number of Robertson four-high mills, with intermediate bright annealing operations. Finishing is carried out on Robertson cold rolling mills of various types which reduce the alloys to the desired dimensions.

One mill worthy of special mention is the Robertson-built Sendzimir mill, which is electronically controlled and has a thickness range of 0·020 in. down to 0·0006 in.,



Cold rolling mill for strip.

Sendzimir electronically controlled strip mill.

Bell type controlled atmosphere heat treatment furnaces.

handling 6 in. wide strip and giving close tolerance on thickness. This is a cluster type mill and precision grinding of roll cambers is essential to successful operation. The work rolls themselves are of very small diameter.

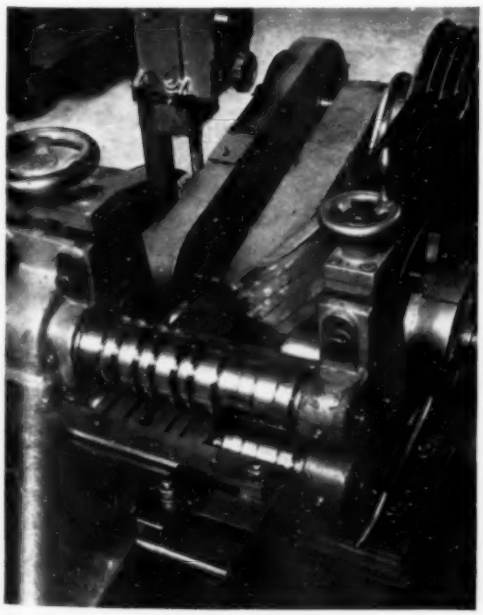
Ancillary Operations

A battery of slitters is available for cutting the strip to widths making it suitable for the production of laminations, cores, etc. Some of the narrower strip is made by roll-flattening of wire. A section of the side bays is devoted to the manufacture of all sizes of cores in Mumetal, H.C.R. and thin Radiometal, mainly for the electrical industry. Another bay is engaged on the production of a wide variety of Mumetal screens in the form of hand fabricated parts and pressings.

The properties of these special magnetic materials are dependent on closely controlled heat treatment and number of bell-type controlled atmosphere furnaces are available for this purpose, using cracked ammonia as the furnace atmosphere.

Rod and Wire Production

For the common types of rod and wire, material received from the cogging mill in the form of $1\frac{3}{4}$ in. squares is handled on the rod and bar mill after reheating. This is a Robertson four-stand three-high cross country mill, whose most common output size is $\frac{5}{16}$ in. diameter. Rod from this mill is coiled on a horizontal axis coiling machine prior to further treatment. For wire drawing, a four-block continuous drawbench is available.



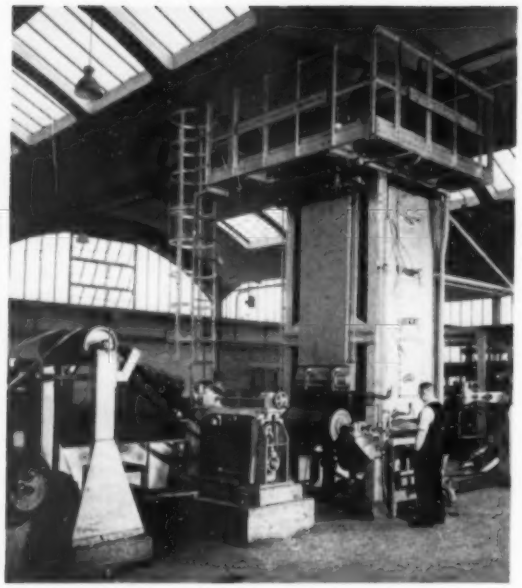
Slitting strip to width.

Inspection and Control

All materials are sampled at the ingot stage and approved on chemical analysis before processing begins. In their final forms, they pass through the Inspection Section, where they are examined for surface defects, magnetic properties and, in the case of beryllium copper, bimetal and special-purpose materials, physical characteristics such as hardness, deflection coefficients and the like. A.I.D. and Admiralty Bonds are under the control of this section, together with a small Admiralty laboratory for the approval of Mumetal rods.

Beryllium Copper Tools

Apart from its use in strip and wire form, where its mechanical properties are of primary importance, beryllium copper is also produced for use in Telcon Bronze safety tools. These may be forged or cast, depending on the tool concerned, and include such varied hand tools as hammers, axes, hoes, picks, pliers, screw drivers, saws, chisels, spanners, trowels and



Continuous Strip heat treatment plant.

wrenches. These tools are non-magnetic and resistant to a number of corrosive agents, and their mechanical properties are comparable with those of steel. Their chief attraction, however, is the fact that, by reason of their comparatively high thermal conductivity, they have little tendency to spark and can be employed with confidence in dangerous atmospheres. It is for this reason that they find application in petroleum refineries, oil installations, chemical factories, paint works, match manufacturers, gas undertakings, explosive manufacturers, distilleries, dust makers, and coal mines.

The beryllium copper foundry occupies part of one of the main bays of the factory, and has facilities for making some 400 different types of tool. In view of increasing demands on the available space, this activity will be transferred to a new 5,000 sq. ft. shop which is in course of erection.

A special beryllium copper alloy containing 2·7% beryllium is available as sand castings or in block form for the production of plastic injection moulds. They are claimed to have better wearing properties than steel; to be capable of taking a superior finish; and to allow more rapid moulding. Apart from machining sand castings, moulds can be made from the alloy by hot hobbing.

Range of Products

Reference was made in the introduction to the early work on soft magnetic materials which marked the Company's venture into the metals field. Since that time the products of the Metals Division have been extended to include electrical resistance alloys, controlled expansion alloys, beryllium-copper alloys, and Elinvar.

Soft Magnetic Alloys

Mumetal is a 77% nickel-iron alloy having high permeability at low field strengths, low hysteresis loss, and low total electrical loss. It is particularly suitable for transformers operating at low energy levels, and for magnetic shields.

Radiometal (50% nickel-iron alloy), with its high initial permeability, high saturation induction, and low electrical losses, has applications in transformers and chokes where the operating flux is higher than that possible with Mumetal, and where a higher permeability than that of silicon iron is desired.

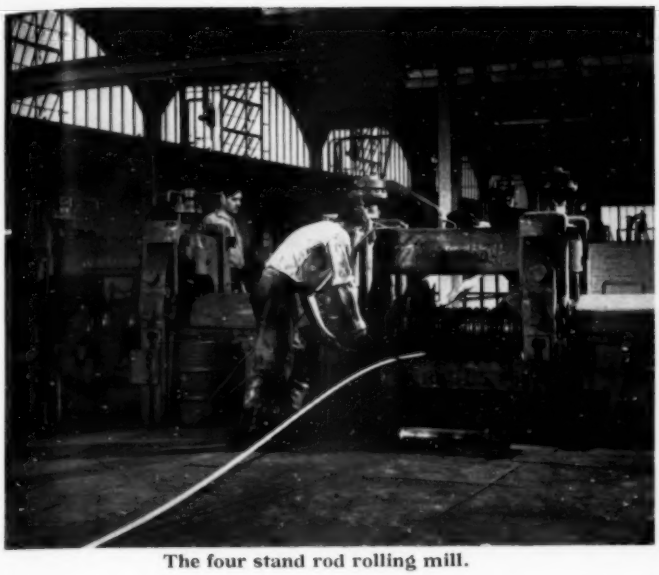
Rhometal has high electrical resistivity and high permeability at carrier telephony and radio frequencies, and finds application in magnetic cores for radar, high frequency, aerial, pulse and television transformers; whilst 36/64 Alloy, with fairly high electrical resistivity, good effective permeability and low electrical loss at high frequencies, is used for high frequency transformers, relay cores and sleeves for quick acting equipment, and screens where a lower performance than that of Mumetal is adequate.

Permendur is an iron-cobalt-vanadium alloy with the highest saturation ferric induction of known materials commercially available. The high flux density which may be developed makes it particularly suitable for rotor and stator lamination in electrical machines for aircraft, and the saving in weight is also of advantage in servo motors. Its excellent magnetostrictive properties are employed in a number of ultrasonic devices.

The 50–50 nickel-iron material—H.C.R. Alloy—specially noted for its rectangular hysteresis loop, is of particular value in magnetic amplifiers and reactors for heavy current mechanical rectifiers, and is also employed in radar transformers and certain telecommunications devices such as frequency multipliers.

The nickel iron magnetic shunt alloy R.2799 has a Curie point at about 60° C. and shows a steady decrease in flux density at a constant magnetising force from −60° C. to +60° C. By the introduction of a magnetic shunt of this nature in certain devices, temperature compensation can be made for variations in flux from permanent magnets and the variation of conductivity of associated copper and aluminium parts.

Pouring beryllium-copper into sand moulds.



The four stand rod rolling mill.

Wire drawing equipment.

Electrical Resistance Alloys

Of the electrical resistance alloys, two—Telcuman and Telconstan—are for use at normal temperatures, and two—Pyromic and Calomic— are for high temperature service. Telcuman is a copper-manganese-nickel alloy for use in precision electrical resistors where minimum change of resistance with temperature is essential, as in standard and sub-standard potentiometers and resistance boxes; whilst Telconstan is a nickel-copper alloy with good mechanical properties and resistance to corrosion, having a fairly constant electrical resistivity over the normal working temperature range of industrial rheostats and semi-precision resistors.

Of the two high temperature alloys, Pyromic, which is of the 80–20 nickel-chromium type, is suitable for service at temperatures up to 1,150° C., whilst Calomic, which contains 65% nickel, 15% chromium and 20% iron, is only applicable where the working temperature does not exceed 1,000° C.

Controlled Expansion Alloys

Bimetal is strip consisting of two layers of metal having different coefficients of thermal expansion and bonded together at the interface. Strips or spirals can be employed to produce deflection or rotation as the temperature changes, and this enables them to be used for the indication of, control of, and compensation for temperature change.

Telcoseal is the name given to Telcon's range of alloys which have special expansion characteristics. They are generally used where a vacuum-tight glass to metal joint is needed, in such applications as radio valves, electric lamps, and cathode ray tubes.

Where minimum expansion is required over the range −50° C. to +200° C., the alloy Invar has long been used. This is made under licence from the original patentees, and has extremely low thermal expansion characteristics.

Beryllium Copper Alloys

Reference has already been made to the use of beryllium copper in the manufacture of tools, but large quantities of an alloy containing 2% beryllium and 0·25% cobalt are supplied for the manufacture of all types of spring, where its high resistance to fatigue and corrosion are of value. Its high resistance to wear when running against steel is a reason for its use for bushings, tappet bearings, and cams.

Elinvar

Elinvar is an alloy employed for precision springs, and in particular those which must remain unaffected by changes in temperature. In addition to the other qualities of a good spring alloy, it has a low coefficient of linear expansion and an extremely small temperature coefficient of Young's modulus. These properties make Elinvar suitable for hard springs in clocks, watches and indicating instruments.

Research and Development Laboratories

In any organisation whose main business is the production of special purpose materials, research and development work are essential to progress, and at the new Telcon Works some 50,000 sq. ft. of floor space have been allotted to laboratories devoted to work in the following fields.

General Metallurgy.—The exploitation of progress in metallurgical science necessitates trials on a pilot production scale, and these laboratories are equipped with this aspect in mind. The plant embraces melting, hot working, cold working, and heat treatment equipment of a very comprehensive nature. Facilities are available for experimental production of new alloys in the form of castings, strip, wire, etc., and the close liaison between these and other laboratories ensures efficient translation of theory into practice.

Physical Metallurgy.—Examinations which may be carried out in this field are very wide in scope, and include some concerned with microstructure and others with crystallographic structure. The comprehensive equipment of these laboratories is in accord with the wide range of alloys that the Metals Division produces.

Making magnetic measurements in the laboratory.

Magnetism.—All D.C. characteristics, including, for example, normal induction curves, hysteresis loops and incremental permeability, can be determined with high accuracy. Facilities are available for the investigation of special characteristics, e.g., the relation between temperature and magnetic quality, direction and magnetic quality, and measurement of flux densities in the region of 25 kilogauss. Properties under A.C. conditions are measured with high precision electronic instruments, and include the magnetisation curve, dynamic loop, power loss and inductance in the range from power to radio frequency.

Electricity.—The laboratories are concerned with investigations to maintain and develop the high quality of precision resistance wires and tapes, and special equipment has been developed for the measurement of very low temperature coefficients of resistance.

Mechanical Properties.—Stress-strain curves, endurance characteristics, hardness and impact strength are investigated in the mechanical laboratories, in which special machines are developed to investigate specific characteristics as investigations require.

Chemical Analysis.—The analytical laboratories are essentially equipped to determine and develop techniques for the determination of not only the standard elements, such as vanadium, beryllium, cobalt, molybdenum, etc., but also less well known elements often added in small quantities to enhance the properties of Telcon alloys. Quantitative spectrographic analytical techniques are employed, as well as absorptiometric, potentiometric, and conventional wet chemical methods.

Physics.—This laboratory handles the investigation of physical characteristics which do not come within the purview of the other laboratories. For example, the thermal expansion of Invar and Telcoseal glass-to-metal sealing alloys; the deflection and force characteristics of the thermostatic bimetals; the high temperature life characteristics of heat resisting alloys; and the particle size and shape of metal powders are all investigated in the physics laboratory.

As a result of the work carried out in the laboratories during the last few years, the following materials are now being produced: (*a*) Super-Mumetal—a magnetic material with the highest permeability of any commercial alloy; (*b*) Special Radiometal—a Radiometal with a greatly improved permeability; (*c*) thin strip for magnetic applications with a thickness down to 0·0005 in.; (*d*) 24 Permendur—a modified Permendur which is more ductile and more readily machined than the standard alloy, although of somewhat lower magnetic quality; (*e*) Vicalloy—a ductile permanent magnet material for instruments where magnets of intricate form are required; (*f*) magnetostrictive alloys; (*g*) Telconal—a resistance alloy comparable with Telconstan but containing no nickel; (*h*) new thermostatic bimetals with enhanced corrosion resistance; and (*i*) 35/18 alloy—a resistance alloy for electrical heating containing a lower proportion of nickel than Pyromic and Calomic.

Mercast Precision Casting

Pending the erection of a new building at Crawley, Sankey-Telcon, Ltd.—a company owned jointly with Guest, Keen and Nettlefolds, Ltd.—is housed temporarily in the administrative block, along with the research and development laboratories. Sankey-Telcon is concerned with the production of precision castings by the Mercast process, and both prototype and production work are being carried on side by side. This is a "lost mercury" process, in which the patterns are made by the solidification of mercury in a master mould. The mercury patterns are then coated with refractory to form the mould, and the mercury allowed to melt and drain from the mould cavity. The merits of the process lie in the fact that the final castings are of such a precision nature that machining is eliminated or greatly reduced. It is also possible to build up complex patterns by cold-welding simple frozen mercury components, and thus to produce finished parts which could not be made by other methods. The output is being eagerly sought by a number of aircraft, radar, and precision engineering firms, and the demands have made necessary, the erection of the new building mentioned earlier.

Metrovick Ex-Apprentices Register

METROPOLITAN-VICKERS ELECTRICAL CO., LTD., is revising its "Register of Ex-Apprentices and Ex-Trainees". The last edition, containing 10,000 short biographies, was published in 1950, and forms asking for present particulars have been sent to all those whose addresses are known to the Company. Any ex-apprentice or ex-trainee who has not received a form (or has not yet replied to one) is invited to write to Mr. R. F. Marshall, Manager, Education Department, Metropolitan Vickers Electrical Co., Ltd., Trafford Park, Manchester 17.

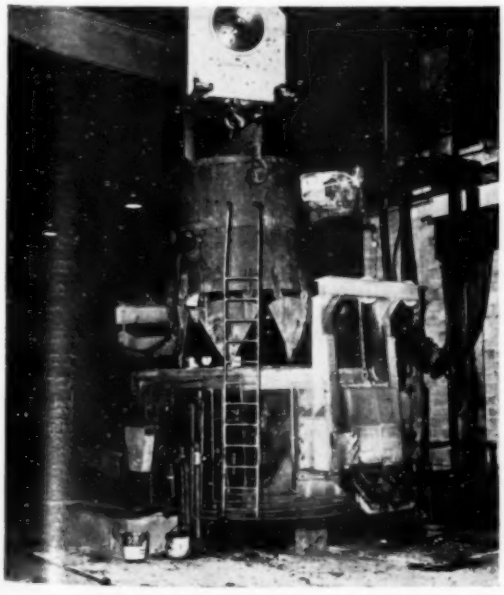
British Melting Furnace in Canada

G.W.B.-Tagliaferri Unit in Alberta

The new furnace in operation.

SINCE the war, Canadian industry has been expanding rapidly, largely owing to the vast new oil fields which have been opened up. As evidence of the spread of industrialisation into areas whose economy was formerly based almost solely on wheat, a new steel works, Premier Steel Mills, Ltd., has recently been opened in Edmonton, Alberta. Premier E. C. Manning officially opened the plant, which took only 10 months to build from the start of work on site to the rolling of the first steel bars.

This new merchant mill is the only steel producer between Winnipeg and Vancouver, and is dependent solely on scrap to keep it going. Formerly, the scrap which the farming industry had available was shipped to the coast or Winnipeg, so that it was not worth the farmer's time to collect his old steel. Premier Steel Mills, Ltd., has now radically altered the situation, and every piece of scrap from the province and surrounding district can now be absorbed. With a length of 600 ft., the plant has an area of 72,000 sq. ft. and cost $2,000,000 to build ; the 3-phase direct arc melting furnace for this whole plant was supplied by G.W.B. Furnaces, Ltd., of Dudley, and is to the designs of the British Company's Italian associates, Leone Tagliaferri & Co., of Milan. The mill is designed for continuous flow production, the steel scrap entering one end and emerging as finished steel bar at the other. The maximum annual output at present is 35,000 tons, and the number of persons employed is over 100.



Charging basket in position over furnace.

The furnace has a nominal capacity of 10 tons with an 11 ft. shell, and is rated at 4,700 kVA., being fed through a 13,600 V., 3-phase, 60 c./s. supply. Following modern practice, the unit is of the top charging type, in which the roof is lifted and swung aside to receive the charge from the baskets brought into position by the shop crane. It is designed for an output of 96 tons per 24 hours, and in actual practice the output varies between 90 and 145 tons per 24 hour working day. Three 12 in. diameter graphite electrodes are provided, the operation of which is controlled hydraulically by means of the Tagliaferri system. This is claimed to be superior to the motor operated method since a finer degree of control and faster electrode operation are obtained, partly owing to the absence of inertia. The electrode control system is part of a common hydraulic system for tilting the furnace body, lifting and swinging aside the roof, and raising the door. A take-off from the hydraulic system permits unclamping of the electrodes to be carried out rapidly, thus reducing to a minimum the time required for working on the hot roof. When in position, the electrodes are held by spring-loaded clamps.

The ingots are between 42 and 60 in. long and 4½ to 5¼ in. square, and the finished product at the present moment comprises flats and bar for concrete reinforcement, the bar being scored on the surface for this

purpose. Future developments will provide for the manufacture of steel sections.

The British part of the project is valued at over $200,000 which, in itself, is a notable contribution to British exports in the hard currency field, apart from representing British equipment in the opening stages of industrialisation in Western Canada.

The next stage in the development and expansion of Premier Steel Mills, Ltd., is now being planned, and as part of these developments a further electric steel melting furnace is to be added. This will be a G.W.B. unit identical with the first, except that the tap changing equipment will be of the "on-load" instead of the "off-load" type fitted initially.

Fully Supported Aluminium Church Roof

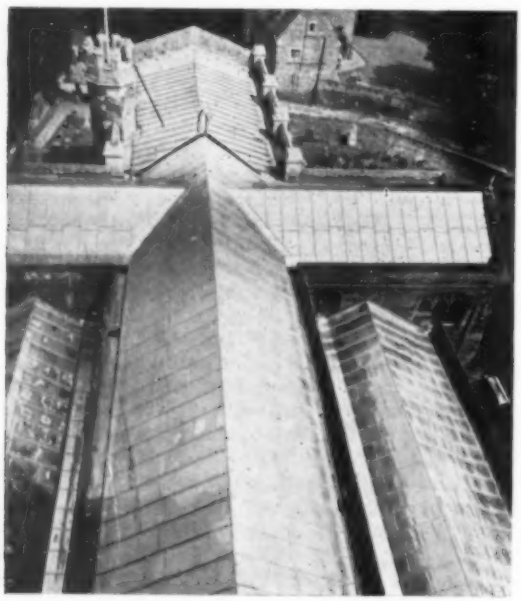
Cost Less than Scrap Lead Realised

AN interesting fully-supported roofing installation, which has recently been completed, is that of St. Mary's Church, Adderbury, Oxfordshire, in which aluminium has been used to replace lead that had deteriorated after many years of service. It is noteworthy that the total cost of the new covering was considerably less than the price obtained for the old when sold as scrap.

The area re-roofed covers nave, aisles, chancel and porches to a total of about 7,000 sq. ft., the lead removed weighing 20½ tons. In its place, over the existing timber boarding, was laid 0·036 in. thick (20 s.w.g.) Noral 3S alloy sheet in the dead soft condition, weighing about 2 tons. This material differs from the 0·028 in. thick (22 s.w.g.) commercially-pure aluminium that has been used in roofing other churches in the same district during the past five years, but being thicker and harder it will reduce possible damage by the boots of maintenance parties, and so will last longer; it also lies flatter and, therefore, looks better. Moreover, although it could not be formed as easily as the softer, thinner material, the requisite folds were made without undue difficulty. The sheet was supplied by Northern Aluminium Co., Ltd., in coils 27 in. wide weighing 56 lb. (corresponding to a length of about 50 ft.) and was laid using the same technique as with copper. The work was undertaken by Builders' Iron & Zincwork, Ltd., the consultant architect being J. M. Surman, Esq., L.R.I.B.A., Architect to the Diocese of Oxford.

A layer of inodorous felt, held in position by aluminium clouts, was first laid over the wood to provide a good measure of sound-proofing. The joints from ridge to eaves were formed as standing seams at 2 ft. centres, and the cross-joints, which were staggered to avoid build-up in the standing seams, were double-lock flat welts at about 5 ft. 6 in. centres. The sheets were secured by aluminium cleats formed in each joint at intervals, and fixed with aluminium screws and nails. Since the existing lead gutters and cesspools were found to be in good condition, they were retained, and where the aluminium sheet made contact with them, or with stonework, it was given a protective coat of bituminous paint.

In addition to the main work of re-covering the roof, fairly extensive repairs were done to the woodwork, all of which was sprayed with an insecticide, and also to the stonework. Yet, with the additional expense of these repairs, the cost of installing the new aluminium roof was over 15% less than the amount realised from the sale of the scrap lead. Such profit, which is expected where aluminium replaces lead as a fully-supported roofing material, is particularly satisfying when it is considered that, in rural conditions, the aluminium roof can be expected to last 100 years.



Hungarian Automatic Foundry

HUNGARY's first fully-automatic foundry at the Budapest Electric Machine and Cable Works has begun test operations. The foundry, said to be "one of the most modern in Europe", will start production early next year. Output from the foundry will be five times greater than normal non-automatic foundry output, and one third fewer workers will be employed: most jobs will be simply push-button ones. The foundry has three cupola furnaces, with an annual output of 11,000 tons each, used in rotation. Engineers are now perfecting ventilating equipment to improve working conditions. German, French and Russian plans for foundry mechanisation were consulted by Hungarian engineers before erecting the foundry, and the experience gained will be used in the planning of other foundries scheduled to be built in the next five years.

Change of Telephone Number

BRITISH INSULATED CALLENDER'S CABLES, LTD., announce that the telephone number of their Exeter Branch has been changed to Exeter 66018-9.

Mechanical Engineering Research 1955*

The Year at East Kilbride

THE work of the Mechanical Engineering Research Laboratory continues to be concerned with the properties and mechanics of materials; fluid mechanics, including hydraulic machinery; lubrication and wear; mechanisms, engineering metrology, and noise control; the formation and machine shaping of materials; and heat transfer and applied thermodynamics. Two new laboratory buildings, for mechanisms and metrology and for heat transfer, are being built, but delays in completing them and in starting new buildings are causing grave concern to the Board.

Hydraulic Machinery

The hydraulic machinery building—the largest and most advanced of its kind in the world—has been designed for fundamental and applied research in fluid mechanics applied to mechanical engineering problems. Constructional work has been completed and most of the equipment has now been installed. The facilities include open circuits for performance tests on water-turbines and pumps, and for calibrating flow-measuring instruments, at flow rates up to 12,000 gal/min.; and a closed circuit for cavitation research on pumps up to 350 h.p. The closed circuit for cavitation research on water-turbine models, now being built, will make it possible, for the first time, to test high-head axial-flow propeller turbines at the same head as the full-scale installation.

Tests on models of pumps and turbines are also being made using air instead of water. One advantage of using air is that wooden or sheet-metal models, which can be easily modified, may be used in the early stages of testing to provide information about flow patterns. The equipment available includes a wind tunnel for air tests on water-turbine models up to 20-in. runner diameter: it will accommodate draught tubes up to 9 ft. × 6 ft.

Precision Speed Control

An electro-mechanical and two electrical systems are being used to control the speed of dynamometers in the Laboratory. They are likely to be useful in industrial operations where accurate control of shaft speed is required. In the electro-mechanical system, a differential is driven by the motor which is being controlled and by a synchronous motor. Any variation in the speed of the controlled motor results in rotation of the output shaft of the differential, the direction of rotation depending on whether the motor is running fast or slow. This shaft is connected to a variable rheostat, or to an electric amplifier, to correct the motor speed.

The first electrical system is for continuously variable speeds. A disc on the motor shaft generates 60 pulses a revolution, so that the pulses per second equal the revolutions per minute. The pulse frequency and the output from an oscillator are fed into an electronic frequency comparator which adjusts the power supply to the motor if it varies from the required speed. The other electrical system controls speed in steps, and uses an electronic batching counter. This will count any number of pulses from 2 to 9,999, and give out a pulse when the required number is reached. The counter is set to give one pulse per second at the correct shaft speed and these pulses are fed into an electronic differential together with pulses derived from a quartz oscillator. The electronic system corrects the speed of the motor if it should vary from the desired rev./min. The electrical systems are simpler than the electro-mechanical system, and are accurate to better than 0·01%.

Creep of Struts

In aircraft and guided weapons operating at supersonic speeds, the heating due to air friction may raise the temperature sufficiently to cause creep of structural parts. The basic principles underlying the creep of structures are therefore being studied. In the first place, the creep deflection of struts has been investigated, to see whether it is possible to predict their behaviour from simple creep tests on the material.

Tests have been carried out on a magnesium alloy whose creep properties at room temperature are similar to those of heat-resistant materials at high temperatures. It was found that data from simple creep tests on this material enabled the creep deflection of magnesium alloy struts to be predicted with an accuracy of about 5%, if the deflections were small compared with the length of the strut.

Locomotive Wheel Adhesion

If adhesion between locomotive wheels and rails could be improved, the maximum loads pulled by locomotives might be increased and their acceleration and braking would be more efficient. The Laboratory is carrying out for the British Transport Commission a fundamental investigation of the causes of variation in adhesion between wheel and rail.

Laboratory equipment for the experiments consists of discs, driving and driven, to simulate the rolling and sliding action which takes place in service. The discs are made of the steels which are used for railway wheels and track, and driving disc and driven disc are kept in contact under the pressures which would be encountered in service. The apparatus is enclosed in a cabinet in which varying conditions of temperature and humidity can be produced.

The effect of weather on slipping has also been studied on a local railway. Thousands of tape recordings have been made of the exhaust beats of locomotives travelling up a gradient of 1 in 60. The tape also records time signals from a "speaking clock." Slipping is revealed by an increase in the frequency of the exhaust beats. It has been shown that temperature and humidity have little effect on slipping, but that slipping occurs much more frequently after light rainfall than after heavy rain.

The effect of sand and steam on adhesion are also being investigated.

Radioactive Tracers in Lubrication Research

Radioactive tracers are being used at the Laboratory for the detection of wear by making one of the rubbing surfaces radioactive. Any particles transferred to the

* "Mechanical Engineering Research, 1955," is published by H.M. Stationery Office for the Department of Scientific and Industrial Research, price 4s. 0d. (72 cents U.S.A.) by post 4s. 3d.

other surface can then be detected, even when the material transferred is so minute in quantity as to be undetectable by microchemical and other techniques.

These tracers can also be used for studying the behaviour of lubricants. The Laboratory is at present investigating in this way the suitability of lead sulphide as a lubricant under conditions of high temperature and pressure. In the machine in which the tests are carried out, a rotating cylinder is crossed at right angles by a second cylinder, which is traversed so that the area of contact between the cylinders is continuously changing. Pastes of radioactive lead sulphide are used as lubricant and the thickness of the protective films which form around the areas of contact is measured.

The Efficiency of Power Transmission Units

The first of a range of dynamometers intended to be used for the investigation of power losses in power transmission units is being tested at the Laboratory. This dynamometer, a prototype, has a capacity up to 75 h.p. at a maximum speed of 2,000 rev./min. The special dynamometers will be used in research intended to increase the efficiency of infinitely variable speed devices and gear drives. Refinements built into them to give high accuracy include trunnion bearings with contra-rotating ball races to reduce static losses to a minimum, power supply led in through mercury cups, and vertical earth and ventilating fan leads to reduce interference with the free movement of the motor casing. The torque reactions of the motor and the power absorber are fed to a hydraulic system designed to provide a direct measurement of losses without the need to measure precisely the input and output torques.

Press and Drop Forging

The application of the forging process in industry has depended more on trial and error methods than on a full understanding of the physical processes involved. The Laboratory is studying the mechanics of deformation of metals in compression caused by steady mechanical loading and by impact loading, so that improved techniques may be developed and better and more uniform properties obtained.

Close control of the experimental factors can be obtained if tests are carried out on a model scale, and so work is being done with model drop hammers. Preliminary tests with specimens made of aluminium, lead and copper have shown the conditions under which valid results can be obtained from model tests. The work is yielding information on the type of blow, or series of blows, which is most effective in bringing about the required deformation.

Heat Transfer in Pipes

The efficiency of industrial heat exchange equipment often depends on the rate at which heat flows from a pipe to a fluid flowing through it. The Laboratory is seeking information about heat transfer to fluids ranging from gases to liquid metals. Little is known, for instance, about the effect on heat transfer of bends, changes of section, and valves in the pipe. Experiments on heat transfer at and near bends is now being carried out, and some research has already been done on the effect of changes of section on heat transfer. The local heat transfer coefficient immediately after the change of section is much higher than before it, and the allowance to be made for this in the design of heat exchangers has been estimated.

An important piece of research concerns the heat transfer to liquid metals flowing through pipes, and mercury will be used as the working fluid. The information gained can be applied to the study of the heat transfer to other liquid metals such as sodium, sodium/potassium alloys, lead/bismuth alloy, and lithium.

Open Days at B.I.S.S.A.'s Sheffield Laboratories

THE British Iron and Steel Research Association will hold two 'Open Days' at the Sheffield Laboratories on Thursday and Friday, 6th and 7th September, 1956, for representatives of member firms. Primarily, the 'Open Days' will provide the opportunity for demonstrating the progress which has been made by those Divisions of B.I.S.R.A. housed at Sheffield—Mechanical Working, Steelmaking, and Metallurgy (General)—since the premises were opened in November, 1953, by H.R.H. The Duke of Edinburgh, K.G.

The Mechanical Working Division, for example, will be mounting a series of displays relating to wire-drawing (including high-pressure lubrication), work on forging problems, developments in hot and cold rolling, and studies of sheet metal working characteristics. Steelmaking Division will show various aspects of research into electric-arc furnace operation and economics, advances in ingot feeder-head design, and progress in the immersion-casting technique for steel strip. Open hearth furnace instrumentation will be demonstrated by displays including the B.I.S.R.A. scale model open hearth furnace, equipped with a calorimeter hearth and apparatus necessary for the study of flame characteristics and convective heat transfer. The Division's Process Chemistry Section will display the vertical high-temperature Globar furnace used in the investigation of sulphur pick-up by melting steel from the impingement of oil droplets. Other aspects of the Division's work will be included in the display.

Metallurgy (General) displays relating to electrical sheet research will include work on the effect of impurities on the magnetic properties of silicon-iron, and studies of strain-ageing alpha-iron. Also on show will be exhibits illustrating investigations into grain-boundary properties and their relation to internal friction, and studies of static hysteresis curves for soft ferro-magnetic material. An extensive display of advanced methods of analysis will also be mounted, including micro-chemical, absorptiometric, spectrographic, and vacuum fusion methods.

The many static displays will be reinforced by a series of working demonstrations—notably of such developments as the continuous casting of steel, and the use of automatic gauge control on the 14-in. experimental rolling mill.

While the main emphasis will understandably be concentrated on the work of the Mechanical Working, Steelmaking, and Metallurgy Divisions, those other sections of B.I.S.R.A. located in London, Swansea, and Normanby will also be represented by minor displays. The various services developed by the Association over recent years will also be illustrated, and the associated research organisations housed at Sheffield—the Cutlery Research Council, the File Research Council, and the Coil Spring Federation Research Organisation—will also be represented.

NEWS AND ANNOUNCEMENTS

Welded Structural Design Course

WELDING offers to the structural steelwork industry a means of increasing productivity and improving technical efficiency; a recent Institute of Welding survey shows that about 40% of the industry's output is welded, and that this proportion is increasing. As the use of welding increases, more and more personnel skilled in its technology will be required. One of the most important factors in welding technology is design and, in particular, the design of the connections. The survey referred to above shows that the industry is extremely short of designers and detailers capable of designing good welded connections, and a course is being planned by the Institute to help those who are keen to keep abreast of tecnhical progress to acquire the specialised knowledge which is so important to the designer and detailer of welded structures.

At the end of the course, all students will be supplied with lecture notes and illustrations so that they may compile a handbook of welded structural details.

The first three lectures will deal with fundamental aspects—the arc welding process, the welded joint and the economics of welding design. Then will follow lectures on roof truss details, beam and girder details, column details, rigid frame details, tubular construction, and plate girders. The tenth evening will be taken up by a general discussion with the lecturers.

The course, which will consist of ten evening lectures between October 3rd and December 5th, 1956, will be held at the Junior Institution of Engineers, Rochester Row, London, S.W.1. Further particulars can be obtained from the Secretary, The Institute of Welding, 2, Buckingham Palace Gardens, Buckingham Palace Road, London, S.W.1.

Practical X-Ray Crystallography Course

AN evening course on practical X-ray crystallography will be held in the Physics Department of Battersea Polytechnic commencing on October 9th, 1956. It will be held on Tuesday evenings at 6 p.m., and will consist of approximately 60 hours of lectures, demonstrations and practical work. The course is intended for graduates and others suitably qualified who require practical experience in X-ray crystallography, and will include the operation of gas and vacuum X-ray equipment; the use of standard X-ray cameras; single crystal, powder and back reflection methods; and interpretation of photographs.

The fee for the course is £2, and further details may be obtained from the Head of the Department. Applications for enrolment should be made at least three weeks before the course begins.

Convention of Chemical Sciences 1956

THE Convention of Chemical Sciences 1956 will take place in Paris from 18th November to 3rd December next, at the same time as the IVth Salon de la Chimie—Caoutchouc-Matières Plastiques. The Convention will include several meetings of particular importance, notably the 29th International Congress of Industrial Chemistry, the 1st European Congress of Corrosion, the European Conference of Chemical Engineering and the Paris Technical Meetings.

The International Congress of Industrial Chemistry will hold its sessions from the 18th to 24th November at the Maison de la Chimie. It will consist of 21 sections, which will deal with the main technical problems which are of interest to those industries in which the production is based on chemistry.

The European Congress of Corrosion will also take place from 18th to 24th November at the Maison de la Chimie. Its work will be divided into 8 sections, which will be concerned with all aspects of corrosion and means of combating its effects.

The European Conference of Chemical Engineering will include three symposiums on the following subjects: calculations of converters, calculations of reactors, and extraction by solid adsorbants. These symposiums will take place in the Conference Hall of the IVth Salon de la Chimie, in the Palais des Congress at the Porte de Versailles on 22nd, 23rd and 24th November.

The Paris Technical Meetings will be held at the same place on the following days. They will include a series of specialized symposiums dealing with polar and tropical equipment, plastic materials, natural and synthetic rubber, control and regulation, analysis and methods of test, high-grade and special steels, etc.

Engineering Sandwich Course

WITH the object of providing a theoretical and technological training comparable with that of an engineering degree but so arranged that the student's technological knowledge will advance concurrently with his industrial practical training and provide a qualification acceptable for membership of the Institution of Electrical Engineers and of the Institution of Mechanical Engineers, a sandwich course is being organised at the South East London Technical College, Lewisham Way, London, S.E.4. The course extends over four years, students spending alternate weeks in college and industry. Further particulars may be obtained from the Principal.

Institute of Metals

THE Institute of Metals is now housed in its new headquarters at 17, Belgrave Square, London, S.W.1. Correspondence for the Joint Library and Information Department should, however, still be addressed to 4, Grosvenor Gardens, London, S.W.1.

Government Copper Stocks Disposal

THE Board of Trade are making arrangements for reducing their stocks of copper. This is in accordance with the announcement in the Defence White Paper (Cmd. 9691, paragraph 125) that the United Kingdom's strategic holdings of industrial raw materials would be reduced during 1956/57. The Board propose to release up to 36,000 tons of copper in the current financial year, and have been discussing with trade

representatives how disposals may best be arranged so as to avoid undue disturbance to the market. By reason of the period of notice recommended in the G.A.T.T. resolution of 4th March 1955 on the Liquidation of Strategic Stocks, no sales of copper will be made before the middle of August.

Efco-Edwards Vacuum Metallurgy, Ltd.

A NEW company has been formed jointly by the Electric Furnace Co., Ltd., of Queens Road, Weybridge, Surrey, and the Edwards High Vacuum, Ltd., of Manor Royal, Crawley, Sussex. These two companies specialise in electric furnaces and high vacuum equipment, respectively, and have for some time been working together on vacuum metallurgical problems. The new company, with such experience and resources available, can offer the latest design of equipment for this new field, and enquiries are welcomed at either of the above addresses.

The first products of Efco-Edwards High Vacuum Metallurgy, Ltd., are a range of vacuum melting and pouring furnaces designed for research and production, as well as other standard plant for sintering, brazing, annealing and degassing metals, and larger non-standard furnaces are already at the drawing board stage.

Typical examples of the increased demands made by engineers for metals with improved qualities are to be found in the modern aircraft industry and the rapidly growing atomic energy field. High physical qualities in the special alloys used for aircraft engines and other high duty units without increase in weight or volume can be obtained by producing these materials under vacuum, while atomic energy works using rare metals for reactors already employ vacuum metallurgy on a large scale for processing. Improved characteristics can be given to many well known metals and alloys by treatment under vacuum, and America and Germany, in particular, have proved beyond doubt that there is an important future for this type of plant, which has developed well beyond the experimental and research stage to production units.

University of Nottingham

THE University of Nottingham announces that W. Canning & Co., Ltd., of Birmingham, have established a research scholarship of the value of £600 per annum, and that The Electro-Chemical Engineering Co., Ltd., of Woking, Surrey, have agreed to donate £300 per annum for three years towards the cost of a further scholarship. Both scholarships will be tenable in the Department of Metallurgy, and will be awarded for research in the field of metal finishing.

Nu-Swift American Link-Up

NU-SWIFT, LTD., the manufacturers of fire fighting equipment, announce the conclusion of an agreement between them and Safety First Products Corporation, of Elmsford, New York, N.Y., for Nu-Swift to manufacture under licence and sell throughout the world, except in U.S.A., Canada, Mexico and certain other countries, the patented Safe-T-Meter Dry Powder Extinguisher of advanced design, manufactured and marketed in the North American Continent by the Safety First Products Corporation. The necessary consent of the British Exchange Control has been obtained.

The development of dry powder extinguishers for use on inflammable liquid fires and fires in electrical equipment is not of recent date, but since the war great strides have been made in the design and operational efficiency of these extinguishers, particularly in the United States and Germany.

The manufacture of the extinguishers and the powder will take place wholly at the Nu-Swift factory in Yorkshire. It will, however, be some months before the equipment (the design of which has been further improved by Nu-Swift research engineers), will be available in Britain and in the many oversea markets to which Nu-Swift export, and for which it has obtained the exclusive marketing rights.

Opportunities for Graduates

WHEN the "Directory of Opportunities for Graduates" is published in January 1957, it will be the first and most comprehensive publication devoted to openings for graduates in business and industry. It is to be distributed free to every last-year University student throughout Great Britain, and will contain detailed announcements by leading business organisations setting out the openings available in their firms, the type of graduate they want to employ, and the salary and prospects they have to offer him.

The "Directory of Opportunities for Graduates" was first published in 1955 as a supplement to the handbook "Oxford University What's What", and was also distributed as a separate publication in Cambridge. The success of this pilot edition, which was endorsed by the Oxford University Appointments Committee, and its enthusiastic reception by both undergraduates and advertisers have demonstrated that there is a very real need for a publication of this type. This year, the first 'national' edition of the Directory will contain a preface by Viscount Chandos, Chairman of A.E.I., and a foreword by the Secretary of the Oxford University Appointments Committee. It also has the backing of most of the other University Appointments Boards.

The Directory can in no way usurp the functions of either the Appointments Boards at Universities or the personnel departments of large firms, but is intended to complement them. It is being published to enable graduates to study all the opportunities before choosing a career, and to make their choice with the assistance of industry and commerce, whose personnel problems the Directory at the same time helps to solve.

Quasi-Arc Patent Specification

QUASI-ARC, LTD., of Bilston, Staffordshire, have filed a patent specification for clipping together normal manual arc welding electrodes so that they can be used with the twin-arc welding process. By using clips covered with insulating material, or clips made of an electrically non-conducting material, electrodes which have a conducting or semi-conducting coating can be incorporated.

Change of Telephone Number

THE telephone number of Aero Research, Ltd., Duxford, Cambridge, has been changed to Sawston 2121. The Telex number is 10-101.

RECENT DEVELOPMENTS
MATERIALS : PROCESSES : EQUIPMENT

Automatic Degreasing and Drying

In every section of industry in which metal components are manufactured, the metal surfaces become contaminated with grease, which must be removed before surface coatings can be satisfactorily applied. A convenient method of degreasing which has been employed for some considerable time involves the use of trichlorethylene vapour. This process has the advantage of being simple to control, the work is left in a dry condition and a minimum of space is required.

However, the usual degreasing equipment of the open-top manually operated type has two serious disadvantages :—

(1) There is a loss of the toxic trichlorethylene solvent, as a result of draughts and convection, with consequent high operating costs, and a health hazard for the operator.

(2) The efficiency of the process must depend entirely upon the conscientiousness of the operator. Thus there is always the possibility of under-treatment if the component to be degreased is not held in the solvent vapour for a sufficient time.

There has recently become available the Perlift, a machine which has been especially designed to overcome these disadvantages. It has a totally enclosed construction and an automatic time cycle, and consists of an evaporating tank, with a thermostatically controlled electric heating system, a vapour chamber, and an electrically operated platform hoist. It is available in three standard sizes, with dimensions as follows :—

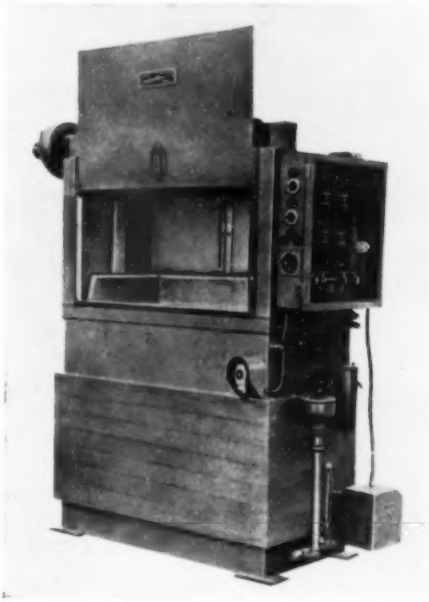
Platform Size	No. 1	No. 2	No. 3
Length	40 in.	60 in.	80 in.
Width	20 in.	25 in.	25 in.
Height	20 in.	27 in.	29 in.
Volume of Solvent ..	3¼ gal.	5¼ gal.	6¼ gal.

Non-standard machines can also be built to order.

The operator has only to place the work on the platform and press the operating button. The sliding door of the machine is automatically closed, lowering the platform into the vapour chamber, where the component resting on it is degreased by the rising trichlorethylene vapour. When the vapour has risen above the level of the work, a temperature-controlled time mechanism causes the platform to rise through the drying zone into the unload position. At the same time, the sliding door opens and a bell signals the end of the sequence.

The degreasing and drying cycles are therefore quite clearly independent of the operator, and are automatically governed by the quantity of work in the machine. No fumes can escape while the Perlift is in operation, as the sliding door is closed. When the door is opened a suction fan comes into operation which prevents the fumes from entering the room.

Perlift machines are manufactured in Switzerland by Technochimie S.A., Zurich, and are imported by The Electrochemical Engineering Co., Ltd., of Woking, who provide the technical staff to supervise installations and service the machines. A stock of essential spares is held in this country.



The Electro-Chemical Engineering Co., Ltd., Forsyth Road, Sheerwater Trading Estate, Woking, Surrey.

Foam Fire Fighter

THE development of an entirely new type of self-contained, portable foam fire fighting appliance has been announced by The Pyrene Company. The new appliance, Model A.F.30C, is a trolley-mounted unit of 30 gal. capacity which produces more than 400 gal. of fire-smothering mechanical foam for use against outbreaks involving quantities of oils, spirits, fats and other inflammable materials. It has been designed to meet a wide variety of fire risks, and has many distinct advantages over the more widely-used chemical foam engine. It is particularly suitable for use where water mains do not provide sufficient pressure for other forms of mechanical foam-making equipment. Additional advantages are that it can easily be manoeuvred and operated by only one man, and that re-charging can be completed on the spot within a few minutes.

A 30-gal. cylindrical tank which comprises the main body of the unit contains a pre-mixed solution of water and Pyrene foam compound. Discharge pressure is provided by a cylinder of CO_2 gas, which is connected to the main container by a short length of flexible high pressure hose. The CO_2 cylinder is fitted with a sealing disc and a lever-operated piercing head. A syphon tube fitted in the tank is coupled to a 30 ft. length of hose which is carried on a hose saddle clamped to the main

container. Foam is generated at the delivery end of the hose by a specially designed miniature Pyrene foam-making branchpipe. The unit is operated simply by unwinding the hose and pulling the CO_2 lever in an upward direction. Total discharge is completed in two minutes. The A.F.30C maintains its efficiency in a wide range of temperatures.

The unit is mounted on a two wheeled trolley of light, tubular steel construction, and is designed to operate in the upright position. The unit weighs 670 lb. when charged, is 4 ft. 11 in. in height and 2 ft. 7 in. in width overall.

The Pyrene Co., Ltd., Great West Road, Brentford, Middlesex.

Low Shrinkage Film

THERE has arisen an ever-growing need in various graphic arts and industrial fields for a sheet film having a dimensional stability even greater than that obtainable with films having the usual low-shrink cellulose safety base. In recognition of this need, Kodak have introduced Kodalith Ortho Sheet Film PB, which is ideally suitable for four-colour reproduction work and in other cases where close register is necessary.

The new film has the well-known regular Kodalith Ortho Film emulsion and non-curl anti-halation backing, but has the added feature of being coated on a polystyrene safety base 0·005 in. thick. This offers unique advantages where size-holding is of critical importance, for the polystyrene base is so dimensionally stable that it would require a temperature change of 20–25° or a relative humidity change of between 30–40% to produce a dimensional variation of less than 1 part in 1,000.

One of the main causes of shrinkage in film is evaporation of solvent and/or plasticiser. Since polystyrene base contains neither of these constituents, shrinkage is virtually non-existent. Polystyrene has a very low moisture absorption, and the size change in processing is, therefore, extremely small (less than 0·02% in either direction) and with negligible difference from one direction to the other. Moreover, the film remains perfectly flat during local re-touching, when parts of the film may be in a moist condition over prolonged periods.

The physical and optical quality of the base is excellent, it is clear and transparent and compares favourably in strength and flexibility with standard cellulose safety base.

The Graphic Arts Division, Kodak, Ltd., Kodak House, Kingsway, London, W.C.2.

Endothermic Generator with Automatic Ratio Control

IPSEN INDUSTRIES, INC., is now offering a unique endothermic atmosphere generator featuring automatic dew point control of the base generator gas. It is a compact unit combining a gas-fired endothermic generator with the Dewtronik automatic dew point recorder-controller.

By interlocking this controller with the generator, the prepared atmosphere gas is controlled within a narrow dew point range by automatic regulation of the air/gas ratio to the reaction tube. The result is continuous, fully automatic control of the prepared base gas analysis under such conditions as changing humidity and variations in analysis and pressure of the line gas. Close control of the air/gas ratio to the reaction tube minimises the tendency to over-supply hydrocarbon, which would be deposited on the catalyst, thus considerably reducing the number of periodic burn-outs and maintenance. This accurate control also makes it possible to produce uniform atmosphere gases with low dew points, and completely eliminates the necessity for manual adjustments and periodic dew point checks.

The new Automatic Endothermic Generator will be available in standard sizes ranging from 750 to 5,000 cu. ft./hr.

Ipsen Industries, Inc., 715 South Main Street, Rockford, Illinois.

CURRENT LITERATURE

Book Notices

SPECTROCHEMICAL ABSTRACTS VOL. IV, 1946–1951

By E. H. S. van Someren and F. Lachman. Royal Octavo, 180 pp., 827 abstracts, bound in limp cloth. 1955, Hilger & Watts, Ltd., 98 St. Pancras Way, London, N.W.1. 30s. net.

THE 827 papers abstracted in Volume IV brings to 1,689 the total dealt with in the four volumes, which cover the period 1933 to 1951. The papers—which include those dealing with emission spectroscopy and flame spectroscopy, in its analytical applications, but not with absorption spectrophotometry, fluorescence spectroscopy or microwave spectroscopy—have been selected, as far as possible, from those recording more than the mere results of spectrochemical analysis and contributing in some way to the development of the subject.

Volume IV follows the well-tried pattern of the earlier volumes. The first part comprises an index of authors, listing the titles of their papers with cross references to the abstracts, and an index of the elements contained in the papers. The second part consists of the abstracts, arranged alphabetically under a series of appropriate sub-headings. The separate sections are devoted to papers on analytical procedure for various substances, apparatus, methods, source theory, reviews and summaries, wavelength tables and atlases, and books. The papers on procedure are further subdivided into those dealing with biological materials, ceramics and slags, gases, oils, liquids, minerals, non-metallic solids, and metals and alloys.

CORROSION AND ITS PREVENTION AT BIMETALLIC CONTACTS

10 pp., foolscap size. Published by H.M. Stationery Office, London, 1956. 1s. 6d. net.

THIS 10-page booklet has been published by H.M.S.O. on behalf of the Corrosion and Electrodeposition Committee of the Inter-Service Metallurgical Research Council (Admiralty and Ministry of Supply). It is an attempt to provide up-to-date and reliable information on the subject, primarily for the benefit of designers in government departments and government contractors, although it should be of considerable interest and value to all who are, or should be, concerned with metallic corrosion.

The text provides general information about the conditions in which bimetallic contacts are most likely to lead to troublesome corrosion, and also about the means of mitigating such effects. This should be read in conjunction with the table which follows: by a system of symbols it indicates the relative effects on the corrosion of a range of metals when brought into mutual contact and bridged by water containing an electrolyte. The table is arranged in two main groups of metals, the first in sequence approximately similar to the "Schedule of Potentials" included in many documents. The second group comprises chromium, stainless steel, titanium and aluminium, which metals develop highly protective oxide films, and behave as relatively noble metals under conditions favourable to the maintenance of such films, but are in some cases less resistant in conditions in which breakdown of the films is possible. It is these metals in particular that cause difficulty in attempting to predict the behaviour of bimetallic contacts from a schedule of potentials.

B.C.A.S. HANDBOOK OF PNEUMATIC EQUIPMENT

210 pp. Published by the British Compressed Air Society, 32, Victoria Street, London, S.W.1.

THIS new edition of the B.C.A.S. handbook explains the characteristics, features and uses of a wide variety of compressed air equipment—such as air and gas compressors, paint spraying equipment, pneumatic tools, etc. Written in an easily understood style, it is particularly valuable to buyers and purchasing agents, as well as for use for training and reference purposes. The present edition incorporates a completely revised 16-page buyers' guide, containing a full list of names and addresses of members and associate members, and a classified list of products from which the manufacturers of over 250 separate items of compressed air equipment can easily be ascertained.

Trade Publications

A CATALOGUE recently issued by Barlow-Whitney, Ltd., describes a range of their smaller and portable industrial heating equipment with wide applications in many industries ranging from radio to rubber, from plastics to printing, and from aircraft to artificial silk. The appliances, which include glue pots, wax pots and droppers, plastic dip pots and tanks, immersion heaters, hotplates, tinning pots, metal pots, soldering irons, ladles, muffles, furnaces, ovens, kettles, wax tanks, and air heaters are all electrically heated, and have automatic thermostatic controls to ensure correct working temperatures. Copies of the brochure are obtainable from Barlow-Whitney, Ltd., Sales Dept., 2, Dorset Square, London, N.W.1.

DURING the last few years, the Steel, Peech and Tozer Branch of the United Steel Companies, Ltd., has developed schemes for the education and training of technical and commercial apprentices. These schemes have attracted considerable attention, and information concerning them has now been published in booklet form as a guide to all boys who have taken General Certificates of Education and who are wishing to embark on careers in engineering, metallurgy, fuel and commerce in the steel industry.

PUBLICATIONS No. 76 and 77, issued by F. A. Hughes & Co., Ltd., deal with the various aspects of the Guardion service and Guardion cathodic protection equipment. The former discusses the theory of corrosion and protection, and outlines the Guardion service which extends from consultation, through site investigation and installation, to an after-sales maintenance service. In applications to buried and submersed structures, cathodic

protection operates under the conditions where normal maintenance may be impossible, and Publication No. 77 is concerned with cathodic protection for marine applications.

DEVELOPMENTS in metal cutting techniques must of necessity have their parallel in progress in cutting fluids and coolants, and in a 64-page brochure, Fletcher Miller Ltd., set out the cutting fluids available, the work they may reasonably be expected to do, and their general characteristics. The brochure is divided into nine sections, the first six dealing with soluble oils, sulphurised straight oils, sulpho-chlorinated straight oils, non-sulphurised straight oils, special duty oils, and paste cutting media. The seventh section deals with sundries for the machine shop, including auxiliary products for water softening and conditioning, germicidal preparations, a hand cleaner and hand cream. Some notes on the employment of cutting fluids to achieve maximum efficiency, including mixing, application, welfare, storage and handling, are presented in section eight, whilst section nine provides general information on the Fletcher Miller organisation, together with an index to the rest of the booklet.

THE BK Fifty Grabbing and Lifting Cranes featured in the Blaw Knox, Ltd., Publication 213/1, can be fitted with an independent power boom hoist which permits the boom to be raised or lowered under mechanical power whilst a load is in suspension. An optional extra attachment is the power hook lowering device which ensures precision lowering by mechanical power under positive control, and enables variable rope speeds to be obtained through adjustment of the throttle control. The brochure gives a comprehensive specification of the machine, including radius and load diagrams.

THE June, 1956, issue of the *Wild-Barfield Heat-Treatment Journal* contains articles on "Mains Frequency Induction Heating for Reaction Vessels and Pipelines" and "Continuous Electric Furnaces." Reference is also made to the Company's exhibits at the International Machine Tool Exhibition, a new 3 kW induction heating unit, a mains frequency induction heater for metal bands, and a chain conveyor billet heating furnace.

REFERENCE was made in a recent issue to the establishment of a Special Alloys Division of Dewrance and Co., Ltd. The Company has recently issued Publication No. 682 dealing with "Endewrance" High Alloy Castings, which are designed for service under extreme conditions of heat, abrasion, corrosion and erosion. Particulars are given of the range of alloys and their physical properties. They can be supplied "as cast" or machined to suit customers individual requirements. Existing facilities include sand moulding, centrifugal equipment, shell moulding and the CO_2 process.

THE National Foundry Craft Training Centre is a non-profit making organisation providing extra training for apprentices in the foundry industry, which broadens and adds to what they are taught in their works, particularly in view of the fact that many foundries are rather specialised in their products. A booklet has recently been published describing the Centre, and copies may be obtained by foundries interested in the scheme on application to the Secretaries, Heathcote and Coleman, 69 Harborne Road, Edgbaston, Birmingham, 15.

AT an exhibition at Dorland Hall, London, some ten years ago, Johnson Matthey & Co., Ltd., attempted to give an idea of the Company's range of work. Since that time, the field of the Company's activities has become enlarged to meet new and growing demands, and a beautifully illustrated booklet has recently been issued to give an idea of the Company's work today. This includes the refining and marketing of precious metals for bullion and for the jewellery and allied trades; the production of high purity chemicals and spectrographic standards; the production of special industrial alloys, mainly based on the precious metals, and of resistance and contact materials; and the production of materials for the decoration of pottery and glassware, and of pigments for the paint, plastics and rubber industries.

THE June, 1956, issue of *The Bonderizer*, published by the Metal Finishing Division of Pyrene, Ltd., deals with the application of the Division's protective coatings in three fields. These are: the drawing office, as represented by photo copying machines made by E. N. Mason & Sons, Ltd.; the automobile field, where the Standard Vanguard III also incorporates gears and pinions in the rear axle assembly which have been Parco-Lubrized; and domestic electricity installations, with Bonderized and stove enamelled service units made by British Insulated Callender's Cables, Ltd.

WE have received from the Incandescent Heat Co., Ltd., a reprint of an article on Thermal Engineering by the Incandescent Group, which appeared in *British Machine Tool Engineering*. The Group had its beginning over fifty years ago, when a number of engineers worked together on a new idea in heat treatment furnaces, in which, instead of burning coal in the working chamber, it was burnt in a separate combustion chamber, and the products of combustion led at incandescent heat into the furnace chamber. From this historical beginning, the article goes on to trace the growth of the Group during the last fifty years, to describe briefly the manufacturing facilities, and to conclude with a well illustrated section dealing with some interesting Incandescent installations.

How high strength parts can be manufactured by the use of powder metallurgy processes is shown in a brochure just issued by Machine Products, Ltd., Kembrey Street, Swindon, Wilts. This, the first publication to explain the new processes since their recent introduction at the Company's Swindon factory, places emphasis on the versatility of application. Parts can be produced to a high degree of accuracy and to intricate shapes, virtually eliminating the need for subsequent machining operations and thus freeing machine tools for other urgent work. One of the advantages of the process is that high strength components can be manufactured with most of the characteristics of a casting or forging.

Books Received

"The Casting of Steel." Edited by W. C. Newell. 599 pp. inc. index and numerous illustrations. London and New York, 1955. Pergamon Press, Ltd. 105s. net.

"Steels for the User." By R. T. Rolfe. Third Edition, Revised and Enlarged. 399 pp. inc. index and numerous illustrations. London, 1955. Chapman and Hall, Ltd. 45s. net.

"Plastics for Corrosion-Resistant Applications." By R. B. Seymour and R. H. Steiner. 423 pp., inc. author and subject indexes. New York and London, 1955. Reinhold Publishing Corporation and Chapman and Hall, Ltd. 60s. net.

LABORATORY METHODS
MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC
INSTRUMENTS AND MATERIALS
AUGUST, 1956 Vol. LIV, No. 322

A New Etching Reagent for Steel and Cast Iron

By B. V. Guellard, B.Sc.

Research Laboratory of The Steel Company of Wales, Abbey Works, Margam.

An account is given of experience with a new etching reagent for steel—sodium bisulphite solution—which has proved superior to some of the etchants in everyday use. Apart from its value as a general etching reagent, it can also be used to reveal phosphorus rich areas and chemical segregation at the grain boundaries.

A COMMUNICATION from I.R.S.I.D. has been published on the application and usefulness of a new etching reagent for steels—sodium bisulphite. This reagent is claimed to be superior in its attack on a polished steel surface to many other known etchants used in everyday practice, and also to be very easy to control. The merits of sodium bisulphite solution have been investigated in the author's laboratory, and the results obtained were very satisfactory.

It is claimed by the investigators, L. Beaujard and Mlle. J. Tordeux, that the new reagent belongs to the class which comprises solutions used as tint etchants. This type of etchant reveals not only the boundaries of grains, but also indicates their crystallographic orientation. The advantage of sodium bisulphite is that it can be used at room temperature, instead of at the high temperature necessary for other etching reagents applied for the same purpose. The results to be reported in this article go beyond the work so far reported by the French investigators.

Summary of Original Paper[1]

The idea of an investigation into the application of sodium bisulphite for etching metallographic samples evolved from some indications that this reagent would attack steel. All the possible concentrations and conditions were examined systematically, and a graph of concentration of the solution against time of attack was plotted. Some observations regarding the etching are written across the different fields of the diagram, Fig. 1, which is taken from the paper by Beaujard and Tordeux.

From this representation of the results, one learns that the best action is obtained with a 60–80% aqueous solution of the standard 35% sodium bisulphite solution, and that the optimum time of attack varies from 10 to 25 seconds. The reagent of standard concentration, as used by the French investigators, was an aqueous solution of "technical quality" sodium bisulphite, with a specific gravity of 1·32–1·33 (35°–36° Baumé), containing 35% of the bisulphite. No satisfactory effect could be obtained when the reagent was used at temperatures higher than 30° C. The investigation involved about 300 samples, etching being effected at room temperature. Each sample was polished electrolytically or chemically before it was subjected to the action of the reagent.

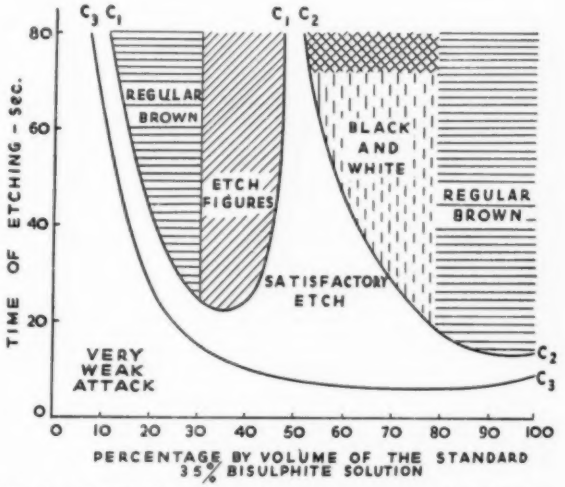


Fig. 1.—Effect of solution strength and time of etching on results obtained

Other fields on the diagram which may be of some interest are those of 30–45% and of 70–80% of the standard solution. In the former case, etch figures are indicated when the attack is longer than 25 seconds, and in the latter the etch is expected to be more 'contrasty' (black and white grains) for an etching time longer than 40 seconds. Sometimes the white grains which have not been fully attacked show square etch figures, which indicate that the surface of the specimen cuts through such grains in a plane closely approximating to the (100) plane.

Referring further to the application of the reagent, the following other recommendations have been made: (*a*) the concentration of the solution should be tested before use; (*b*) if mechanical polishing of the samples is carried out, it must be done very carefully, as the reagent will reveal scratches which would have otherwise remained unnoticed; and (*c*) samples are immersed in the solution for etching, and the attack can be observed with the naked eye.

The photomicrographs and their interpretation found in the original paper were most interesting, and the difference in the effect on microstructures which were

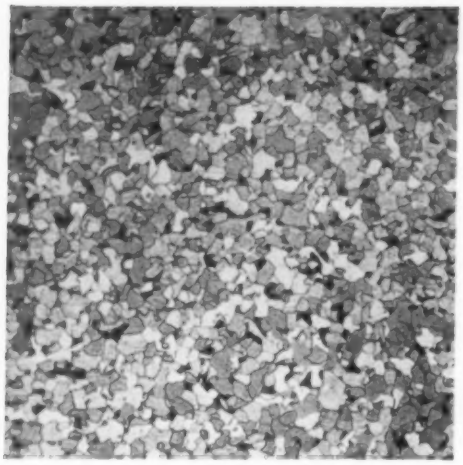


Fig. 2.

Fig. 2.—Longitudinal section of Cor-ten steel. Etched 20 seconds in 100% sodium bisulphite solution.* ×100

* See p. 96 for definition of 100% sodium bisulphite solution.

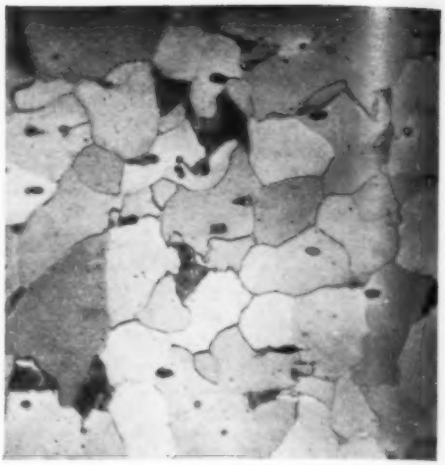


Fig. 3.

Fig. 3.—The same sample as Fig. 2. ×500

Fig. 4.

Fig. 4.—Medium carbon rimming steel showing a band of inclusions in the centre of the cross-section. As polished. ×100

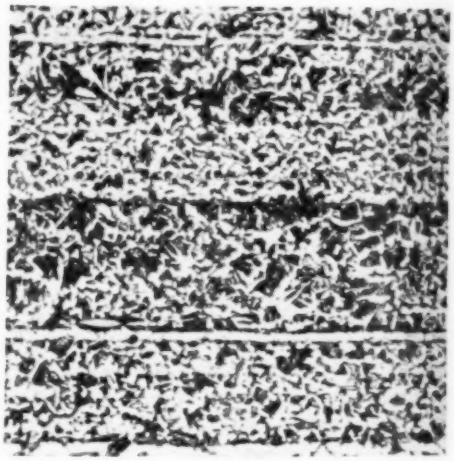


Fig. 5.

Fig. 5.—The same sample as in Fig. 4. Etched in 5% nital. ×100

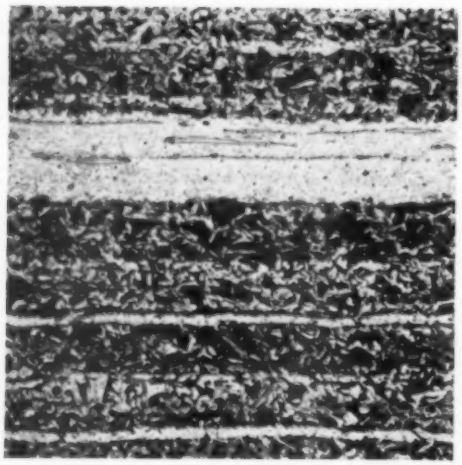


Fig. 6.

Fig. 6.—The same sample as in Fig. 4. Etched in Stead's reagent. ×100

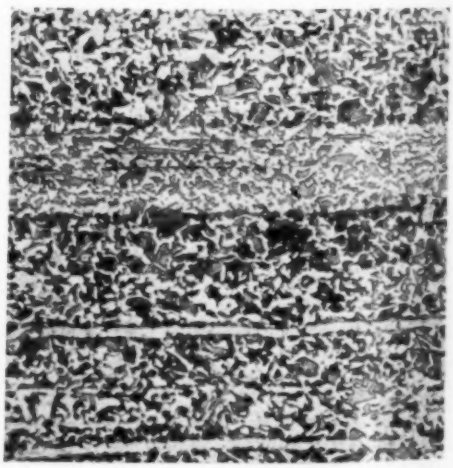


Fig. 7.

Fig. 7.—The same area as in Fig. 6. Etched 10 seconds in 60% sodium bisulphite solution. ×100

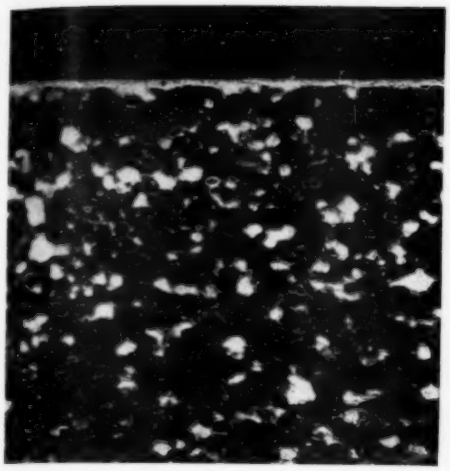


Fig. 8.—Low carbon rimming steel after cold reduction and subcritical annealing. Etched 60 seconds in 100% sodium bisulphite solution. ×100

Fig. 8.

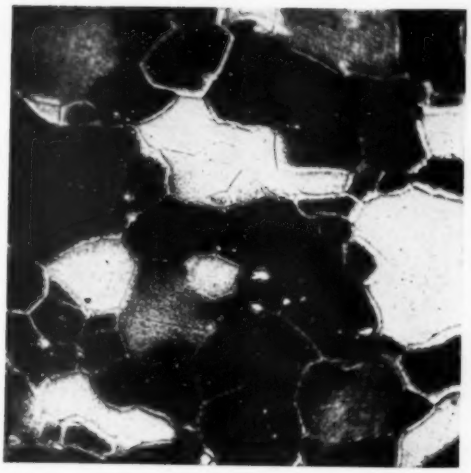


Fig. 9.—The same sample as Fig. 8. ×500

Fig. 9.

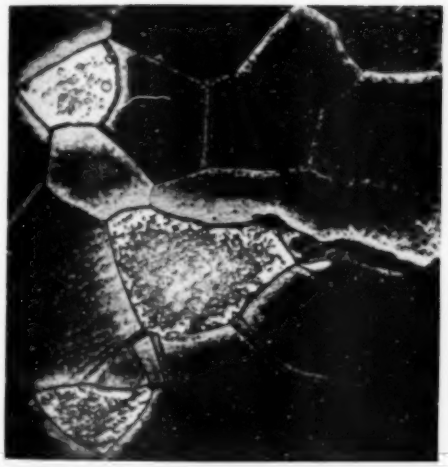


Fig. 10.—A sample of low carbon steel. Etched 60 seconds in 100% sodium bisulphite solution. ×1,500

Fig. 10.

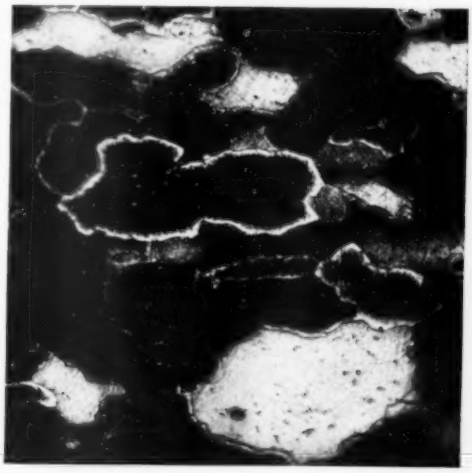


Fig. 11.—Stabilised low carbon steel after cold reduction and subcritical annealing. Etched 60 seconds in 100% sodium bisulphite solution. ×500

Fig. 11.

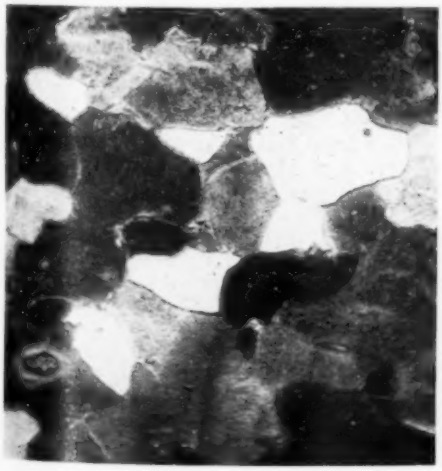


Fig. 12.—The same sample of rimming steel as in Fig. 9 after normalising at 930° C. ×500

Fig. 12.

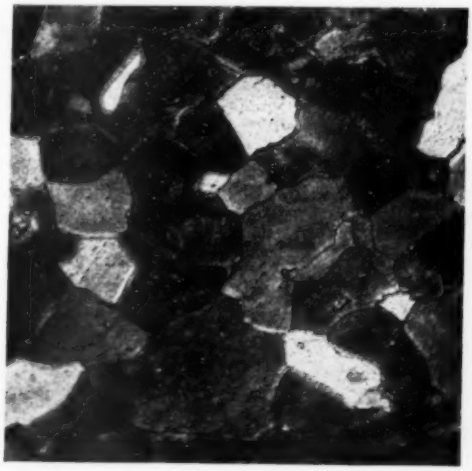


Fig. 13.—The same sample of stabilised steel as in Fig. 11 after normalising at 930° C. ×500

Fig. 13.

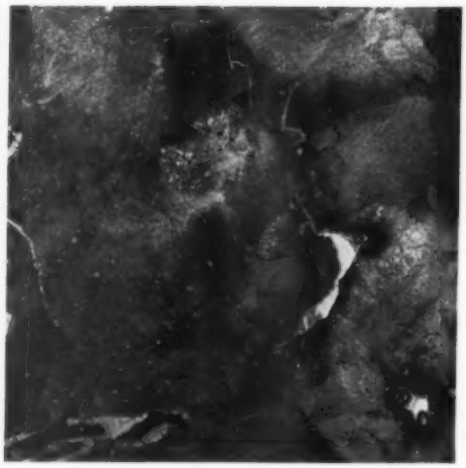


Fig. 14.

Fig. 14.—Grey cast iron showing lines of cementite and grains of phosphide eutectic, with dark areas rich in phosphorus. Etched 20 seconds in 100% sodium bisulphite solution. ×100

Fig. 15.

Fig. 15.—A grain of phosphide eutectic. Etched as in Fig. 14. ×750

obtained by using normal etching reagents and a solution of sodium bisulphite was quite striking. A sample of low carbon Bessemer steel after etching with a 3% nital showed strongly attacked grain boundaries and no tinting. The same steel after etching in sodium bisulphite solution was characterised by grains of different shades of grey, depending on their crystallographic orientation: this type of etch can facilitate grain counting. The austenitic grain structure in a steel containing 1·5% C, 2·5% Cr, soaked at 1,092° C. and quenched in water, looked much more impressive after application of the bisulphite; the austenitic grain size was very clearly defined. The martensite needles became black, whilst the carbides remained white as a result of the etching attack.

Lines of deformation in broken test pieces can be revealed by a saturated solution of picric acid in alcohol, but the features of the microstructure were shown more clearly after an attack with sodium bisulphite. In a photomicrograph taken at a higher magnification, two systems of parallel lines could be seen in some of the grains. These were probably planes of slip. It is thus evident that a sodium bisulphite solution can be very helpful in detecting whether a fracture is of a brittle nature or of a ductile type.

Another set of photomicrographs illustrated how chemical segregation in low carbon Bessemer steel was shown up more clearly by using the new reagent.

Sodium bisulphite solution does not attack cementite and leaves it white. It can, therefore, be regarded as better than the alkaline sodium picrate solution.

The coalesced cementite in a sample of steel was shown to be much more obviously identified when etched with sodium bisulphite solution. The same applied to cementite in a sample after a McQuain-Ehn test.

Results of Further Investigation

After this resumé, the author wishes to add some of his own results and to comment on his experiences with this new etching solution. He would like especially to point out the newly discovered usefulness of the sodium bisulphite reagent for the study of grain boundaries. All samples used for this investigation were prepared by mechanical polishing, and were finished by hand. A standard solution of the reagent called 100% was obtained by dissolving 35 g. of sodium bisulphite in 100 ml. of distilled water. The etching was carried out at room temperature for different periods of time and each sample was rinsed first with water and then with methylated spirits before drying. The effects of etching can be seen in the photomicrographs presented here. It should be realised that structures shown in these photomicrographs may not look as impressive as they were seen under the microscope. There is thus a need for interpretation of the microstructures and for comment on the quality of the etching.

An etched sample of Cor-ten* steel showed tinting of the surface; three different colours of grains could be distinguished. These were evenly distributed throughout the whole surface, and in Figs. 2 and 3 they are visible as different shades of grey.

To demonstrate another property of sodium bisulphite solution, which can be described as sensitivity to chemical segregation, a sample of medium carbon rimming steel was used. This contained a central band with an excessive amount of non-metallic inclusions (Fig. 4). An etch with 5% nital showed no difference between this particular band and the remainder of the steel (Fig. 5), but etching with Stead's reagent showed strong phosphorus segregation (Fig. 6). This segregation can also be observed in the microstructure revealed by etching with the new reagent (Fig. 7).

The most interesting property of sodium bisulphite solution is illustrated in the photomicrographs Figs. 8–13. Fig. 8 is a photomicrograph of a sample of low carbon rimming steel after cold reduction and sub-critical annealing. Apart from random orientation of the grains, narrow white bands can be seen at the grain boundaries. These are also featured in Figs. 9 and 10. Fig. 10 is a photomicrograph at ×1500 of another sample of low carbon steel. Examination of the structure at high magnification showed the bands to be slightly raised from the surface.

In order to investigate the nature of these borders, samples of the same steel were normalised, and, as can be seen from Fig. 12, the borders disappeared almost

* High corrosion resistance steel containing: C, 0·09%; S, 0·03%; Mn, 0·40%; Ni, 0·18%; Cu, 0·3%; Cr, 0·93%; Si, 0·38%; and P, 0·09%.

Continued on page 106

A Non-Destructive Microchemical Method of Sorting Steels

By W. R. Nall and R. Scholey, A.Met., A.I.M.

Bragg Laboratory, Naval Ordnance Inspection Department, Sheffield.

A microchemical procedure is described for the rapid sorting of carbon and low alloy steels which uses simple, inexpensive apparatus. The classification of unknown steels is based on the estimation of manganese, nickel, chromium and molybdenum. A small number of analysed steels are used as standards for comparison purposes. The portable apparatus is self-contained and non-destructive in use, and may be applied to field work.

IT is often required to decide quickly the composition of a number of steels without recourse to lengthy chemical analysis. If the problem is to sort samples which have been accidentally mixed, an accurate chemical analysis is not justified. Existing non-destructive methods include spectrographic analysis and magnetic/electrical sorting, which are capable of giving accurate results, but involve the use of relatively elaborate apparatus. Spot testing techniques on the other hand are purely qualitative, indicating the presence or absence of an element.

The proposed method aims at a compromise, and provides results which are a reliable guide to composition. The apparatus required is easily constructed and the necessary skill in its use is readily acquired. The question of portability and simplicity of operation under field conditions has been considered, because many of the problems occur in stores and yards containing extensive and widely-dispersed stocks.

The elements manganese, nickel, chromium and molybdenum were chosen as a basis for classification tests, since they are of general application to sorting problems. From preliminary work on other alloying elements (copper, cobalt and titanium), it would appear that the scheme could be extended to include these if required.

Electrographic Sampling

Anodic Solution

The flow of a current through an electrolyte or any conductor is a flow of electrons from negative to positive (cathode to anode), and the generator of electricity, e.g. a battery, consists essentially of a negative pole tending to emit electrons with a negative charge, and a positive pole tending to absorb them. If these poles are joined by a conductor, the electron stream flows from negative to positive in accordance with these tendencies. If the conductor is an electrolyte, such as an acid solution, the electrons reach the cathode from the negative pole of the battery, pass through the solution to the anode and so reach the positive pole of the battery. The electrons are carried through the solution by solute particles—ions. There is an opposite flow of positively charged particles, which consist of ions originating in the anode. Hence the anode goes into solution in the electrolyte. Electrographic sampling techniques depend upon this principle of anodic solution.

Existing methods

A survey of the literature showed that this technique had been used for sorting procedures, and had certain obvious advantages, such as portability and speed of operation.

Clarke and Hale[1] described an electrographic method for the qualitative analysis of stainless steels, and suggested that paper chromatography would separate inorganic elements in the electrogram. They thought that this might lead to a comparative test for distinguishing between austenitic 18–8 stainless steels and low chromium-nickel steels.

Long[2] developed an electrographic method for identifying high temperature alloys, and gave details for the rapid detection of chromium, nickel, cobalt, molybdenum, tungsten, titanium and iron, where these elements occurred as major alloying constituents.

Two systematic schemes have been devised, one by Zech[3] for identifying fourteen elements in corrosion-resistant metals, the other by Hughes[4] to distinguish between ten types of plating.

In all the above references, the element to be determined occurred as a pure metal or a major constituent. The same observation applied to the method of Calamari *et al.*[5] for gold in plating, and to his method[6] for chromium in stainless steel. In the latter method, the minimum amount of chromium which could be detected was approximately 10%.

The only electrographic method which could detect a minor constituent in steel was that of Calamari, Hubata and Roth[7] for molybdenum, in which 0·2% could be determined using the reduced molybdenum thiocyanate complex. In this paper[7] the authors noted that :—

> "The intensity of the colour produced increases with the molybdenum content when the current density and time remain constant."

In the discussion of Clarke and Hale's[1] paper one author stated that :—

> ". . . it was doubtful whether an assessment of the quantity of an alloying metal could be made by the method described in their paper. If a vigorously standardised procedure were adopted, i.e., if the time of electrolysis, pressure of electrode, current density, thickness of paper and so on, were made the same, then by comparing electrograms from standard samples of known composition with the electrograms of the test samples a semi-quantitative result could be obtained."

The development of electrographic methods for steel sorting has been delayed by the lack of a standardised procedure referred to above, and also by the mutual interference of alloying elements.

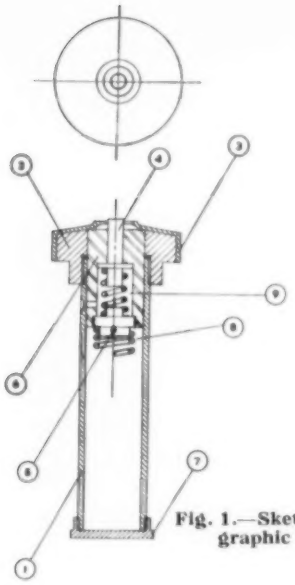


Fig. 1.—Sketch of electrographic sampler.

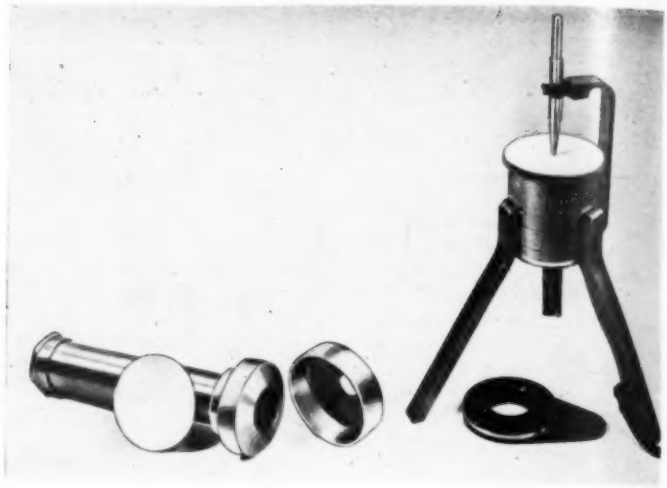


Fig. 2.—Left: electrographic sampler; right: Weisz ring oven.

These two difficulties have been overcome by (*i*) the use of a sampler modified from the type described by Monk[8], and (*ii*) the introduction of a new piece of microchemical apparatus—the Weisz "ring oven".[9] The following description of these two pieces of apparatus and the methods of using them will show how they can be combined to give a versatile sorting procedure for ferrous or non-ferrous alloys. Details will then be given for the analysis of low-alloy steels.

Electrographic Sampler

Fig. 1 shows the details of the sampler, which is designed to use 5·5 cm. filter paper discs in the sampling head. The other components are similar to those described by Monk[8], and comprise a battery case (1) which holds a 3-volt pocket torch battery. The battery must be inserted with the small positive pole pointing outwards, i.e., in contact with the screwed cap (7). This makes the case electropositive, and during the sampling operation the sample is made the anode. When the sampler is pressed against a flat surface, the graphite counter electrode (4) is forced against the filter paper in the sampling head by the spring (9). This ensures that the pressure is reproducible from sample to sample, provided the surface is flat over a circular area approximately 1 in. in diameter. The spring holder (5) is insulated from the case by the plastic cylinder (6), which is held in place by a small screw. An aluminium cap (3), which slides over the head of the sampler (2), centres the filter paper over the counter electrode. Fig. 2 shows, on the left hand side, the sampler with the aluminium cap removed ready for the insertion of a filter paper.

Operation of Sampler

A 5·5 cm. Whatman No. 20 or No. 1 paper is placed inside the aluminium cap, so that it lies flat on the aluminium surface, and the cap replaced in position over the head of the sampler.

The centre of the filter paper is then moistened with an electrolyte. A sufficient quantity must be applied to moisten the area of filter paper covered by the counter electrode; a slight excess is not detrimental if care is taken to prevent the electrolyte from spreading to the cap. The volume required is approximately two microlitres (0·002 ml.), and this amount is conveniently delivered from a piece of glass tubing drawn out to a capillary tip.

The sampler is then pressed firmly against the cleaned metal surface and held in position for a specified length of time. The pressure is then released, the cap removed and the filter paper with the electrolysed sample solution at the centre is taken from the cap. Sample papers prepared in this manner may be dried and stored for subsequent analysis, or the paper may be transferred directly to the ring oven.

Ring Microanalysis[10]

If an attempt is made to identify alloying elements in a drop of oxidised steel solution by placing it on a filter paper and adding selective reagents, it soon becomes apparent that separation of iron is necessary. This may be achieved by treating with a drop of ammonium hydroxide solution, when iron together with any manganese or chromium is precipitated. Nickel and molybdenum remain in solution and may be washed away from the precipitate by a second drop of ammonium hydroxide solution. Further washing of the precipitate to obtain complete separation causes the nickel and molybdenum to be diffused over a large area of the paper. If a selective reagent is now applied to the paper, e.g., an alcoholic solution of dimethylglyoxime, in the presence of nickel an uneven pink stain is obtained which is unsuitable for comparison purposes.

This difficulty is overcome by limiting the area in which the drop can spread and simultaneously concentrating the eluted salts in a small area of the paper. This two-fold advantage is obtained by the use of the Weisz ring oven.

The Weisz Ring Oven[9]

The apparatus is shown on the right of Fig. 2, and consists of a cylindrical aluminium block (5·5 cm. diameter, 5·0 cm. high), supported on three legs and having a central hole 2·2 cm. diameter. A concentric

hollow space in the base of the block accommodates a nickel chromium alloy heating element, which operates on a 50-volt A.C. supply from a transformer. The temperature of the upper surface of the block is adjusted to approximately 110° C. by means of a variable resistance in the circuit. A brass support above the heating block carries a glass guide tube accurately centred above the hole in the block. This is for supporting the capillary pipettes used for applying solutions to a filter paper lying on the aluminium block. The capillary pipettes are prepared from glass tubing of 0·5 mm. bore and 2 mm. wall thickness, and each is approximately 10 cm. long. The tubing is drawn out to a tip 2 mm. in diameter and the end is ground flat.

Operation of the Ring Oven

A 5·5 cm. filter paper with the electrolysed sample solution at the centre is transferred from the electrographic sampler to the top of the heated ring oven, and held flat by means of a ring made of porcelain or other inert material. Reagents and wash solutions are applied to the paper by means of a capillary pipette, which is supported in a vertical position by the guide tube directly above the centre of the filter paper. The tip of the pipette rests on the surface of the paper and the rate of flow of liquid is controlled by the capillarity of the paper. Successive washes of appropriate solutions are applied until all the soluble salts are eluted from the centre of the paper and concentrated in a ring at the boundary of the hot zone, where vaporisation of the solvent takes place. With pipettes of the above-mentioned dimensions, the rate of flow of liquid through the paper is equal to the rate of evaporation at the hot zone, resulting in a narrow ring of deposited salts. The paper is then removed from the ring oven and dried in a current of hot air. The element present in the ring can then be identified by spraying with suitable reagents.

Investigation of Colorimetric Reagents

Feigl[11] recommended several reagents for the colorimetric detection of microgram quantities of manganese, nickel, chromium and molybdenum. It was found, however, that several of these reagents were not sufficiently selective for ring microanalysis. In the detection of chromium, particular difficulty was encountered in the stabilisation of the coloured rings on the paper.

Manganese

The use of an ammoniacal solution of silver nitrate was found to be unsatisfactory, because it reacted with several of the elements present in steel. Benzidine proved to be unsuitable due to its comparatively high limit of identification (0·15 μg.). The most suitable reagent investigated was tetra-base (tetramethyldiaminodiphenylmethane), which had the low identification limit of 0·001 μg. The blue oxidation product obtained was stable for sufficient time to allow comparison with standards.

Chromium

Benzidine gave blue-coloured rings with varying amounts of chromium, but the results were not quantitative. The use of an alcoholic solution of diphenyl carbazide was complicated by atmospheric oxidation of the reagent. Whilst investigating the possibility of using the coloured chromium compounds themselves, the chromates of lead, barium, and silver were produced in the paper. Silver chromate was found to be the most intensely coloured salt, and was selected as the most suitable for the estimation of chromium.

TABLE I.—WEIGHT OF SAMPLE REMOVED FROM TWO STEELS DURING 20 SECONDS ELECTROLYSIS USING DIFFERENT ELECTROLYTES.

Electrolyte	Sample *	Micrograms of Iron Removed †
40% v/v Nitric Acid (6·4N) ..	Carbon Steel	133 ; 125
40% v/v Nitric Acid (6·4N) ..	Low Alloy Steel	144 ; 125
N Hydrochloric Acid	Carbon Steel	39 ; 45
N Hydrochloric Acid	Low Alloy Steel	39 ; 40
0·5 N Hydrochloric Acid ..	Carbon Steel	20 ; 21
0·5 N Hydrochloric Acid ..	Low Alloy Steel	21 ; 23

* Analysis of Samples :—

	Mn%	Ni%	Cr%	Mo%
Carbon steel	1·5	0·18	0·12	—
Low alloy steel	0·71	2·81	0·65	0·52

† Determined colorimetrically.

Molybdenum

The method described by Calamari *et al.*[7], which involved the formation of a molybdenum thiocyanate complex and its subsequent reduction, was found to be suitable for the ring oven method.

Nickel

The complex formed between nickel and dimethylglyoxime could be precipitated in the paper, and was found to give stable rings which proved satisfactory for comparison purposes.

Analysis of Carbon and Low-Alloy Steels

Preparation of Samples

Remove rust, scale and any foreign matter from a circular area of approximately 1 in. diameter of the sample surface.

If a large number of samples are to be examined, a portable grinding machine will be found useful. Where no power supply is available, or where a smaller number of samples are to be prepared, filing is satisfactory. The surface finish must give an even contact with the filter paper during sampling. Remove any oil or grease from the cleaned surface by wiping with a cloth soaked in a suitable solvent.

Choice of Electrolyte

Several electrographic sampling techniques[5,6,7] employed salt solutions as electrolytes, but it was found that the salts accumulated in the ring at the boundary of the hot zone and tended to mask the colour reaction. This was particularly noticeable with the small amounts of an element removed from a steel which contained only 0·1–0·2% of the element concerned.

Dilute acids were used to overcome this disadvantage, and Fig. 3 shows the current/time graphs for various electrolytes used. The amount of sample removed during electrolysis can be controlled by varying the strength of the electrolyte (Table I). Small amounts of an element may be determined by increasing the strength of the electrolyte until the amount of element present in solution falls within the useful detection range of the particular reagent.

During anodic solution of a steel using hydrochloric acid as electrolyte, the elements iron, manganese, nickel and chromium enter solution in their lowest valency states. The behaviour of molybdenum under these conditions has not been established. When nitric acid is used as electrolyte, according to Calamari *et al.*[7], iron dissolves as Fe^{3+} and chromium as chromate CrO_4^{2-}.

With small amounts of chromium, however, there is no evidence of the presence of chromate formed during electrolysis using various strengths of nitric acid as electrolyte. The following electrolytes have been chosen as most suitable for the estimation of the respective elements :—

Manganese	0·5 N hydrochloric acid
Nickel	0·5 N hydrochloric acid
Molybdenum ..	1 N hydrochloric acid
Chromium	6·4 N nitric acid (s.g.1·20)

Estimation of Manganese

As previously stated, during anodic solution of a steel using hydrochloric acid as electrolyte, ions of iron and manganese are present with double positive charges (Mn^{2+} and Fe^{2+}). A separation is effected by treatment with ammonium hydroxide in the presence of ammonium salts. The ferrous hydroxide first precipitated is oxidised by atmospheric oxygen to ferric hydroxide and forms a brown precipitate, together with any chromium present. Under these conditions, manganese remains in solution and is oxidised to the heptavalent state (MnO_4^-) with potassium periodate. The heptavalent manganese salts are then treated with a solution of tetramethyldiaminodiphenylmethane to give an intensly coloured blue oxidation product.

REAGENT SOLUTIONS

Electrolyte (*0·5N Hydrochloric Acid*).—Dilute 5 ml. hydrochloric acid (s.g. 1·16) to 100 ml. with water.

0·5% *Potassium Periodate Solution*.—Dissolve 0·5 g. of potassium periodate in 50 ml. of hot water, cool, and dilute to 100 ml. with cold water.

0·5% *Tetra-base Solution*.—Dissolve 1 g. of citric acid in 1 ml. of cold water, add 0·25 g. of tetra-base (tetramethyldiaminodiphenylmethane). Stir until dissolved and dilute to 50 ml. with water.

2% *Ammonium Chloride Solution*.—Dissolve 2 g. of ammonium chloride in water and dilute to 100 ml.

0·1 *N Hydrochloric Acid* (*approx.*).—Dilute 20 ml. of 0·5 N hydrochloric acid to 100 ml. with water.

PROCEDURE

Prepare a sample paper with the electrographic sampler, electrolysing for 20 seconds and using 0·5 N hydrochloric acid as electrolyte. Remove the paper from the sampler and allow to dry (Note 1). Expose the paper to ammonia fumes for 10 seconds and transfer to ring oven. Wash the precipitate once with 0·1 N hydrochloric acid, using a capillary pipette, and then seven times with 2% ammonium chloride solution. Allow the paper to dry on the ring oven for 1 minute, then remove and spray with 0·5% potassium periodate solution. (An all-glass atomiser should be used for spraying the paper with reagent. The spray produced must be composed of small droplets only, to ensure even wetting of the paper). Dry the paper (Note 1) and spray with 0·5% tetra-base solution. Compare the intensity of the blue-coloured ring with rings prepared in a similar manner from steels of known manganese content (Note 2). The blue colour fades slowly, and each batch of samples must be compared with appropriate standards prepared at the same time. Comparison must be made within 5 minutes after spraying with the tetra-base solution.

Estimation of Nickel

As previously stated, after anodic solution of steel using hydrochloric acid as electrolyte, ions of nickel and iron are present with double positive charges. A separation is effected by treatment with ammonium hydroxide. Ferrous hydroxide first precipitated is oxidised by atmospheric oxygen to ferric hydroxide. Basic nickel chloride is also precipitated, but is soluble in excess ammonium hydroxide to form a nickel-amino-chloride which reacts with an alcoholic solution of dimethylglyoxime to give an intensely coloured red precipitate.

REAGENT SOLUTIONS

Electrolyte (*0·5 N Hydrochloric Acid*).—Dilute 5 ml. of hydrochloric acid (s.g. 1·16) to 100 ml. with water.

75% *v/v Ammonium Hydroxide Solution*.—To 25 ml. of water add 75 ml. of ammonium hydroxide (s.g. 0·880).

1% *Dimethylglyoxime Solution*.—Dissolve 1 g. of dimethylglyoxime in 100 ml. of industrial ethyl alcohol (methylated spirit).

PROCEDURE

Prepare a sample paper with the electrographic sampler, electrolysing for 20 seconds and using 0·5 N hydrochloric acid as electrolyte. Remove the paper from the sampler and allow to dry (Note 1). Transfer to the ring oven and wash eight times with 75% ammonium hydroxide solution, using a capillary pipette. Allow the paper to dry on the ring oven for one minute, remove and spray with 1% dimethylglyoxime solution and expose to ammonia fumes. Allow the alcohol to evaporate and compare the intensity of the red ring with rings prepared in a similar manner from steels of known nickel content.

The nickel-dimethylglyoxime complex is stable, and a series of standard rings prepared from steels of known nickel content may be kept for reference.

Estimation of Chromium

Chromium and iron are present as chromate and ferric ions, respectively, after anodic solution with nitric acid as electrolyte. Reduction to trivalent chromium occurs in the presence of the filter paper, re-oxidation to the hexavalent state being effected with an ammoniacal solution of hydrogen peroxide. Green chromite ions are first formed by reaction with excess alkali hydroxide, and in the presence of an oxidising agent these are quantitatively oxidised at room temperature to chromate ions. Highly-coloured silver chromate is then precipitated in the paper by treatment with silver nitrate.

REAGENT SOLUTIONS

Electrolyte (*Nitric Acid* (*s.g.* 1·20)).—Dilute 40 ml. nitric acid (s.g. 1·42) to 100 ml. with water.

Hydrogen Peroxide/Ammonia Solution.—Mix equal volumes of hydrogen peroxide (10 vol.) and concentrated ammonium hydroxide solution (s.g. 0·880).

5% *Silver Nitrate Solution*.—Dissolve 5 g. of silver nitrate in water and dilute to 100 ml.

PROCEDURE

Prepare a sample paper with the electrographic sampler, electrolysing for 20 seconds and using nitric acid (s.g. 1·20) as electrolyte. Remove the paper and dry (Note 1). Transfer to the ring oven and wash twice

with the ammoniacal hydrogen peroxide solution, then four times with distilled water. Allow the paper to dry on the ring oven for 1 minute, then remove and spray with 5% silver nitrate solution. Allow the paper to dry in the air and compare the intensity of the yellow coloured ring with rings prepared in a similar manner from steels of known chromium content.

The yellow coloured rings are stable for 15 minutes when exposed to light, but they can be kept for periods of an hour or more if stored in a light-tight box.

Estimation of Molybdenum

Ions of molybdenum after anodic solution in hydrochloric acid appear to exist as MoO_4^{2-}. Separation from divalent iron is effected by treatment with ammonium hydroxide solution. Ferrous hydroxide first precipitated is oxidised by atmospheric oxygen to ferric hydroxide. The soluble molybdate is treated with an ammonium thiocyanate solution, followed by reduction with stannous chloride to form an intensely-coloured orange thiocyanate complex.

REAGENT SOLUTIONS

Electrolyte (*N Hydrochloric Acid*).

75% *v/v Ammonium Hydroxide Solution.*

Ammonium Thyiocyanate/Stannous Chloride Solution.—Dissolve 10 g. of ammonium thiocyanate in water and dilute to 100 ml. Dissolve 2 g. of stannous chloride in 1 ml. hydrochloric acid (s.g. 1·16) warming slightly to obtain a clear solution. Cool and dilute to 100 ml. with water. Mix equal volumes of these two solutions before use.

20% *v/v Hydrochloric Acid.*

PROCEDURE

Prepare a sample paper with the electrographic sampler, using 1 N hydrochloric acid as electrolyte and electrolysing for 20 seconds. Remove the paper and allow to dry (Note 1). Transfer to the ring oven and wash five times with 75% ammonium hydroxide solution. Allow the paper to dry on the ring oven, then remove and spray with the ammonium thiocyanate/stannous chloride solution, finally spraying with 20% hydrochloric acid. Compare the intensity of the orange-coloured ring with rings prepared in a similar manner from steels of known molybdenum content.

The molybdenum thiocyanate complex is stable for approximately 5 minutes after reduction, but comparison should be made as soon as possible after spraying.

Combined Estimation of Nickel and Molybdenum

In the separation of nickel from iron, molybdenum accompanies nickel in solution, and is eluted with it to the ring. It is thus possible to estimate the two elements on a single sample. The nickel-dimethylglyoxime complex is destroyed by treating with hydrochloric acid, and the molybdenum thiocyanate complex is formed after reduction with stannous chloride.

REAGENT SOLUTIONS

Electrolyte (*N Hydrochloric Acid*)—See manganese reagents.

75% *v/v Ammonium Hydroxide Solution.*

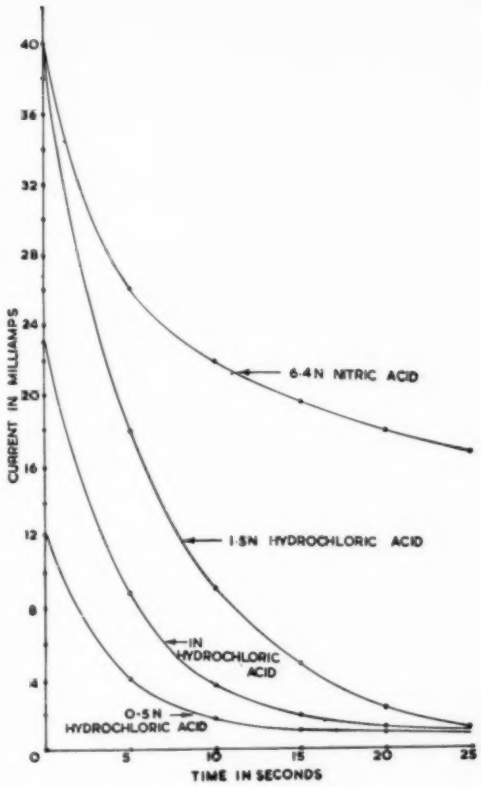


Fig. 3.—Rate of decrease of current for various electrolytes using the electrographic sampler.

1% *Dimethylglyoxime Solution.*—Dissolve 1 g. dimethylglyoxime in 100 ml. industrial ethyl alcohol (methylated spirit).

Ammonium Thiocyanate/Stannous Chloride Solution.—See molybdenum reagents.

PROCEDURE

Prepare a sample paper with the electrographic sampler, electrolysing for 20 seconds and using 1 N hydrochloric acid as electrolyte. Remove the paper and allow to dry. Transfer to the ring oven and wash eight times with 75% v/v ammonium hydroxide solution, using a capillary pipette.

Allow the paper to dry on the ring oven for 1 minute. Remove and spray with 1% dimethylglyoxime solution. Allow the alcohol to evaporate and compare the intensity of the red ring with rings prepared in a similar manner from steels of known nickel content. Spray with 1·5 N hydrochloric acid and dry to destroy the nickel/dimethylglyoxime complex. Spray with the ammonium thiocyanate/stannous chloride solution and compare the intensity of the orange coloured ring with rings prepared in a similar manner from steels of known molybdenum content.

Notes

Note 1.—Sample papers may be allowed to dry in the air or may be dried by heating. In sampling a number of steels, the first sample papers prepared will dry without heating before the sampling of all the steels is complete. If only one or two sample papers are required,

TABLE II.—STEELS SUITABLE FOR STANDARDS.

Sample	Composition by Chemical Analysis				B.S. Specn.
	Mn %	Ni %	Cr %	Mo %	
1	1·5	0·4	0·25	0·40	En 17 (range V)
2	1·3	0·9	0·50	0·20	En 100 (range V)
3	0·56	1·8	0·25	0·25	En 160 (range S)
4	0·71	2·8	0·65	0·53	En 25
5	0·56	4·1	1·25	0·15	En 39B

TABLE III.—COMPARISON OF RESULTS OBTAINED BY RING OVEN TECHNIQUE AND NORMAL CHEMICAL ANALYSIS.

Sample	Mn %		Ni %		Cr %		Mo %	
	Ring Oven	Chem. Anal.	Ring Oven	Chem. Anal.	Ring Oven	Chem. Anal.	Ring Oven	Chem. Anal.
1	1·5	1·7	0·2	0·23	<0·3	0·12	0·4	0·48
2	0·7	0·9	2·5	2·7	0·6	0·57	0·6	0·51
3	1·0	0·79	<0·2	0·22	<0·3	0·15	<0·1	<0·1
4	1·5	1·58	0·2	0·24	<0·3	0·1	<0·1	<0·1
5	0·6	0·67	2·5	2·68	0·75	0·68	0·45	0·40
6	0·5	0·63	2·0	2·08	<0·3	0·09	<0·1	<0·05
7	0·5	0·51	3·0	3·37	0·5	0·8	0·25	0·32
8	0·5	0·48	4·0	4·29	1·0	1·35	0·25	0·25
9	1·0	0·7	0·5	0·78	1·8	1·74	0·35	0·35
10	0·5	0·67	0·9	0·86	1·5	1·57	0·30	0·35

they may be dried in an air oven at 110° C., or by means of a domestic hair drier. A hair drier is also useful in field work for drying papers in the manganese and chromium methods.

Note 2.—The method of choosing standard steels for use in the ring oven method of sorting is best illustrated by an example. If the problem is to decide whether a sample complies with say, a specified manganese content of 0·7–1·0% (B.S.S. En. 43A), two analysed samples of 0·7% and 1·0% manganese content respectively are tested at the same time as the unknown sample. By this procedure the manganese content is readily checked. It is found in practice that only four or five steels are required to serve as standards for the determination of the four elements. A useful selection is shown in Table II, which comprises steels complying with B.S. specifications.

Note 3.—The ring oven and ancillary equipment is available commercially from Perfax Engineering Co., Ltd., Brook House, Torrington Place, London, W.1.[12]

Results

The results of a series of experimental determinations are set out in Table III, with the chemical analysis results for comparison.

Acknowledgment

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The Libraries of Greater Manchester

THE Manchester Joint Research Council's report "Science and Industry" (1954) stated that one-fifth of the firms visited could have solved many of their problems had they known where to go for information. A guide to library resources has recently been published giving all pertinent details of the public, university, newspaper, and industrial libraries of Greater Manchester, many of which are regarded as primary sources of information in their fields. A code number under each entry indicates to what extent the library can be used by the general public, its loan and photocopying arrangements, and many other useful facts concerning its holdings and services. Information officers, research workers and students should find this guide most valuable and timesaving. Copies can be obtained, price 15/- post free, from Mr. G. E. Haslam, B.A., F.L.A., Central Library, Manchester, 2.

Plastics Abstracts Service

STEPS are being taken to make better known overseas the unique service offered by the British Plastics Federation in publishing each month abstracts from technical articles in journals throughout the world dealing wholly or partly with plastics developments. The service includes the publishing of patents from the countries which have an important plastics industry.

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First International Tin Council Meeting

THE International Tin Agreement came into force on July 1st, and the first meeting of the International Tin Council began in London on July 2nd. The Council was welcomed by the RT. HON. PETER THORNEYCROFT (President of the Board of Trade), who pointed out that the International Tin Agreement was the first international agreement since the war which provided for a buffer stock. The Agreement had the advantage of being representative of both producing and consuming countries and, of being compatible with the existing machinery for trading in tin and in particular the international tin market. Very great interest would be shown in the working of the buffer stock which, he hoped, would prove successful in stabilising prices to the common benefit of consuming and producing countries.

The meeting was attended by representatives of Australia, Belgian Congo and Ruanda-Urundi, Belgium, Bolivia, Canada, Denmark, Ecuador, France, India, Indonesia, Federation of Malaya, the Netherlands, the Federation of Nigeria, Spain and the United Kingdom.

Liquid-Liquid Extraction Procedures in Inorganic Analysis

A Review of Practical Applications with Particular Reference to Metallurgical Analysis

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The growing complexity of modern materials has led to difficulties in their chemical analysis resulting from the increasing multiplicity of other substances accompanying the constituent to be determined. The development of new separation methods has made possible the continued use of classical analytical procedures in the face of increasing competition from physical methods, and in the present series of articles the author surveys liquid-liquid solvent extraction techniques in inorganic analysis, with particular reference to metallurgical analysis. In the last of the series, the author discusses the extraction of the platinum metals and miscellaneous extractions.

(*continued from page* 51 *of the July issue of* METALLURGIA)

EXTRACTION OF THE PLATINUM METALS (Pt, Pd, Ir, Rh, Ru, Os)

Platinum

FROM the information available, it would appear that none of the six platinum metals is extractable from nitric acid solutions by diethyl ether. Only trace amounts of platinum enter the organic layer in the ether extraction of 6N hydrochloric acid but, in the presence of a reducing agent such as stannous chloride, ether or ethyl acetate will extract chloroplatinous acid from solutions not less than 1·0N in hydrocloric acid. In contrast to the behaviour of ether, methyl isobutyl ketone extracts 2·0% of platinum[IV] from 2·8N hydrochloric acid, and ethyl acetate removes as much as 2·6%.[23]

Diisopropyl ether extracts only 0·1% of platinum from 2·9N hydrobromic acid, ethyl acetate removes 2·9%, and methyl isobutyl ketone removes 13·5%.[23] Reasoning by analogy, one would expect platinum to be much more readily extracted from hydriodic acid. Kitahara[38] reports, however, that platinum is not extracted by ether from 6·9N hydriodic acid. On the other hand, West and Carlton[40] indicate that platinum is partly extracted from a 5% hydrochloric acid solution containing potassium iodide, when methyl isopropyl ketone is used as solvent. This difference in behaviour of platinum in iodide solutions can almost certainly be attributed to the superior solvent powers of the ketones in general for the halides of the platinum metals. Diethyl ether does not extract platinum from fluoride solution.

Fischer and Bock[42] give no data on the extraction of the platinum metals from thiocyanate solution, but one may infer from the work of West and Carlton that platinum may be extracted as its thiocyanate from acid solutions by use of methyl isopropyl ketone as solvent.

Quadrivalent platinum does not react with dithizone, but in weakly acid solution platinum[II] may be extracted by carbon tetrachloride. Whereas the colour of the aqueous solution is violet-red, the carbon tetrachloride extract is green in colour. Sandell[29] suggests that a colloidal suspension is probably obtained rather than a true solution in the organic layer. The complex is insoluble in chloroform. Platinum[IV] is not precipitated by cupferron from 5% sulphuric acid, and is presumably not extractable as its cupferrate. No information could be found on the behaviour with oxine.

PERCENTAGE EXTRACTION OF THE PLATINUM METALS FROM HYDROCHLORIC AND HYDROBROMIC ACID SOLUTIONS[23]

Solvent	Acidity	Pd	Pt	Rh	Ir	Ru	Os
Isopropyl ether	2·9M HBr	0·16	0·10	0·07	0·03	0·00	25
	2·0M HBr	0·22	0·21	0·15	0·03	0·00	43
Ethyl acetate	2·9M HBr	1·7	2·9	0·16	0·08	0·43	13
	2·0M HBr	1·7	1·7	0·12	0·15	0·43	8
	2·8M HCl	0·31	2·6	—	—	0·62	—
Methyl isobutyl ketone	2·9M HBr	20·5	13·5	—	—	—	56
	2·0M HBr	—	—	0·05	0·10	—	54
	4·0M HCl	1·0	—	—	—	—	—
	2·8M HCl	—	2·0	1·2	1·0	1·3	12

Palladium

Palladium is not extracted by ether from nitric or hydrochloric acids. Isopropyl ether removes only 0·22% from 2N hydrobromic acid, whilst ethyl acetate removes 1·7% and methyl isobutyl ketone some 20%. Under comparable conditions, ethyl acetate extracts 0·3% from 2·8N hydrochloric acid and methyl isobutyl ketone extracts 1% from 4·0N hydrochloric acid.[23] Kitahara[33, 37] found that neither hydriodic acid nor hydrofluoric acid give up palladium to an ether extraction. West and Carlton[40] obtained partial extraction of palladium from a 5% hydrochloric acid solution containing potassium iodide, using methyl isopropyl ketone as solvent and also using methyl isobutyl ketone.[98] West and Carlton, *loc. cit.*, also obtained complete extraction of palladium using methyl isopropyl ketone with an acid solution of palladium containing ammonium thiocyanate.

No information could be found on extraction with cupferron, but the dithizonate may be extracted from dilute mineral acid using chloroform or carbon tetrachloride. The oxinate may also be extracted using chloroform as solvent. Palladium forms chelates with

the 1, 2 dioximes, such as dimethylglyoxime and furyldioxime. The latter complex can readily be extracted into chloroform. Palladium also forms an extractable green complex with *o*-nitrosophenol. Petroleum ether is said to be a suitable solvent. Between *p*H 6·3 and 10·3 palladium may be extracted by capric acid in ethyl acetate.[9] Ruthenium is also extracted, but only in the presence of copper.

Iridium

In contrast to the behaviour of platinum and palladium, iridium is appreciably extracted (5%) by diethyl ether from 6N hydrochloric acid solution. Methyl isobutyl ketone extracts only 1% from 2·8N hydrochloric acid. From 2N hydrobromic acid, isopropyl ether, ethyl acetate and methyl isobutyl ketone extract 0·03, 0·15, and 0·10% of iridium respectively. Iridium has, therefore, a greater tendency to remain unextracted than any of the other platinum metals. Diethyl ether does not extract iridium from hydriodic acid or hydrofluoric acid solution and, according to West and his co-workers,[40, 98] it is not extracted from hydriodic acid solution by either methyl isopropyl ketone or methyl isobutyl ketone. Kitahara[37] quotes 7% extraction of iridium from hydrobromic acid by diethyl ether.

Dithizone does not react with iridium, and no information is available on extraction with cupferron or 8-hydroxyquinoline. Iridium may, however, be extracted into pyridine from aqueous solution.[133]

Rhodium

Rhodium is not extracted from hydrochloric acid solution by diethyl ether, but, in the presence of stannous chloride, ethyl acetate extracts rhodium[III] from aqueous solutions containing a sufficiently high concentration of hydrochloric acid (>2N). According to McBryde and Yoe,[23] diisopropyl ether removes only 0·07% of rhodium from 2·9N hydrobromic acid, while ethyl acetate and methyl isobutyl ketone extract 0·12% and 0·05%, respectively, from 2N hydrobromic acid. Simultaneously, these authors report that methyl isobutyl ketone is a superior solvent for the extraction of the chloride, since it extracts as much as 1·2% from 2·8N hydrochloric acid.

Rhodium was not examined by Kitahara[36, 37] in his study of the extractability of metals from hydriodic and hydrofluoric acid solutions. West and Carlton[40] report that rhodium is at least partially extracted from an acid iodide solution by methyl isopropyl ketone. The extraction is almost complete in the presence of lead. From the same authors, it may be concluded that rhodium[III] is more or less completely extractable by the same solvent from an acid solution containing ammonium thiocyanate.

Like iridium, rhodium may be extracted from aqueous solution by pyridine.[133]

Osmium

Osmium is not extracted from hydrochloric acid (6N) by diethyl ether, but McBryde and Yoe[23] found that methyl isobutyl ketone is effective in extracting as much as 12% of osmium from the 2·8N acid. Whilst none of the other platinum metals is readily extracted from hydrobromic acid, osmium is exceptional in its behaviour. Thus diisopropyl ether extracts 25% and 43% from 2·9 and 2·0N hydrobromic acid solutions, respectively. Ethyl acetate is much less efficient, extracting 13% from 2·9N acid and only 8% from 2N acid. Methyl isobutyl ketone, on the other hand, extracts 56% and 54% from 2·9 and 2·0N hydrobromic acid solutions, respectively. Kitahara[36,37] again reports that neither hydriodic acid nor hydrofluoric acid solutions of osmium give up the metal to an ether extraction. West and Carlton also report non-extraction of osmium from iodide solution by methyl isopropyl ketone.

Hirsch[134] has reported that osmium tetroxide forms a blue coloured compound with potassium thiocyanate in acid solution. This compound may be extracted with ether or amyl alcohol. Thiocarbanilide gives a red-coloured ether-extractable compound which may be extracted by carbon tetrachloride.[136] Extraction with dithizone is ineffective. No information could be found on the possibility of extracting with cupferron or oxine.

Ruthenium

Ruthenium is not extracted by ether from 6N hydrochloric acid, but methyl isobutyl ketone extracts 1·3% from 2·8N hydrochloric acid, and ethyl acetate 0·62%, according to McBryde and Yoe.[23] Diisopropyl ether does not extract ruthenium perceptibly from hydrobromic acid, but ethyl acetate extracts 0·43% from 2–2·9N hydrobromic acid. Data for the extraction by methyl isobutyl ketone are not given. West and Carlton[40] state that while ruthenium is not particularly well extracted by methyl isopropyl ketone from acid iodide solution, it is much more readily extracted along with lead. Kitahara[3, 37] was unable to obtain extraction of the metal from hydriodic acid or hydrofluoric acid by diethyl ether. Sandell[29] states that the thiocyanate may be extracted by butanol, and from the work of West and Carlton[40] it appears that complete extraction may also be obtained using methyl isopropyl ketone.

Dithizone does not form an extractable compound with ruthenium and no information is available on cupferron extraction, but Sandell[29] observes that the oxinate may be extracted with chloroform. As mentioned previously, ruthenium may be extracted along with copper by a reagent consisting of capric acid in ethyl acetate,[9] but in the absence of copper no extraction occurs.

MISCELLANEOUS

Extraction of Mineral Acids

Data for the partition of mineral acids between aqueous solutions and immiscible organic solvents are frequently found in the literature. It is well known that diethyl ether will extract considerable amounts of phosphate from 6N hydrochloric acid solutions. The extraction of acetic and hydrochloric acids by tributyl phosphate has recently been discussed by Pagel, Toren and McLafferty,[137] and many other examples can be cited, but only one paper will be discussed here, and this only because it may well signpost developments in the future.

Smith and Page[138] pointed out in 1948 that long chain aliphatic tertiary amines such as methyldioctylamine have powerful acid binding properties similar to those of the acid binding ion exchange resins. These acid binding powers are due to the insolubility in water of the salts formed with mineral acids. These salts are, however, remarkably soluble in chloroform and similar organic solvents.

When a 50% (v/v) solution of hydrochloric acid was shaken with an equal volume of methyldioctylamine,

an upper oily layer of the hydrochloride and excess base separated out from a lower aqueous layer which had a *p*H of 6·5. The free base has a solubility of less than 0·01% in water and the hydrochloride about 0·5%. The trace of methyldioctylamine remaining in the aqueous layer was recovered by shaking twice with chloroform. The base was recovered by shaking up with aqueous alkali, and was then used for further extractions. Several solvents for the methyldioctylamine were examined, and nitrobenzene and chloroform were chosen as being most suitable.

The results obtained when a known quantity of aqueous acid was shaken with a slight excess of 5% methyldioctylamine in chloroform are shown below. The figures in the last column represent the percentage of the original acid found after re-extraction from the chloroform.

Acid	Normality	Equivalent of Amine Used	Percentage in Aqueous Layer	Percentage Recovered
Acetic	0·1	2·0	3	90
Chromic	0·1	1·5	2	97
Hydrochloric	1·0	1·0	1	98
Nitric	0·1	1·2	0	98
Oxalic	0·1	1·2	4	98
Phosphoric	0·21	1·2	44	50
Succinic	0·37	1·0	11	90
Sulphuric	1·0	2·0	1	98
p-Toluene sulphonic ..	0·05	1·2	3	97

Certain salts, such as that of sulphuric acid, are less soluble than the hydrochloride in chloroform, and tend to separate out as a third layer.

Simple primary and secondary amines are not effective extracting agents. Aromatic secondary amines such as dibenzylamine are not so effective as aliphatic amines with the same number of carbon atoms. The acid binding powers of the tertiary amines improves with the length of the aliphatic chain. The authors suggest that this technique might be useful for the recovery of metals such as chromium and vanadium after oxidation to the anionic form.

Extraction of Phosphorus

The extraction of phosphate as molybdophosphoric acid by various solvents has been the subject of many investigations. The literature on the subject has been thoroughly reviewed by Wadelin and Mellon.[75] These authors point out that, whilst the methods are generally effective, a common drawback is the fact that the presence of any of the other central atoms of heteropoly acids may cause interference, and they examined the possibility of selective extraction of the various heteropoly acid complexes. Solvents which have been used to extract molybdophosphoric acid are ethyl ether,[139] ethyl acetoacetate,[140] ethyl acetate,[141] butyl acetate,[142] 2-methyl-1-propanol,[143] 3-methyl-1-butanol,[144] *n*-octanol,[145] mixtures of butanol and chloroform, etc.[90, 91] Hure and Ortis[146] separated molybdophosphoric acid from molybdosilicic acid by extraction of the former into ethyl acetate. Other workers used butyl acetate for the same separation.[142] Molybdophosphoric acid is selectively extracted in the presence of silicon and arsenic by a butanol-chloroform mixture[90,91] and, according to Wadelin and Mellon *loc. cit.*, the procedure can be extended to treating the residual aqueous solution with a mixture of butanol and ether to remove molybdoarsenic acid, and finally with butanol to extract molybdosilicic acid.

As a result of their study, Wadelin and Mellon concluded that esters are the most selective extractants for molybdophosphoric acid, l-butyl acetate being the best. The 25% solution of butyl alcohol in chloroform suggested by Filippova and Kuznetsova[93] gave highly selective extraction, and the use of chloroform had the advantage of minimising the extraction of various other ions, e.g., $FeCl_3$. They decided, however, that a 20% solution was preferable, since the more concentrated reagent was subject to more interference. The procedure was successfully applied to the determination of phosphorus in steels.

Separation of Cyanide and Thiocyanate

In developing a method for the colorimetric determination of cyanide and thiocyanate in presence of each other, Kruse and Mellon[147] found it necessary to separate the two. The procedure developed was by solvent extraction, and is therefore noted here. It is based on the extraction of the cyanide as hydrogen cyanide from acetic acid solution by isopropyl ether. The ether layer is washed twice with small portions of 5% acetic acid and the washings are discarded. The cyanide is back-extracted into an aqueous solution by shaking the ether layer with 3% sodium hydroxide.

Diethyl ether was first examined, but even though a sixfold volume was used and the aqueous phase was acidified with several acids, the best recovery obtained was 93% and reproducibility was poor. Thiocyanate was extracted as ferric thiocyanate when iron[111] was present. The extraction of cyanide by an approximately threefold volume of isopropyl ether gave 95% recovery and the results were reproducible. Ferric thiocyanate was not extracted by this solvent. Investigation of several acids, e.g., acetic, citric, hydrochloric, phosphoric, and sulphuric acids, revealed that the extraction was not only a function of *p*H, but also depended on the acid used. Thus, more cyanide was extracted at *p*H 2·5 from a solution acidified with acetic acid than from a solution acidified to *p*H 0·5 with the other acids. The optimum extraction occurred from 5% acetic acid. Further increase of acidity merely favoured solution of the sample in the ether layer without affecting the efficiency of the extraction.

It may be added that the thiocyanate was determined by adding pyridine and copper sulphate to the aqueous solution and extracting the thiocyanate complex with chloroform in the *p*H range 2·5–4. The colour or absorbency of the copper complex in the chloroform layer was then measured. Solvents such as *o*-dichlorobenzene and butanol, i.e., solvents possessing some dipole, were also used to extract the complex, but they were inferior to chloroform. Carbon tetrachloride did not extract the complex. A fivefold excess of cyanide could be tolerated at the 1 p.p.m. thiocyanate level. Amounts in excess of this were destroyed by boiling the acid solution.

Separation of Bromine and Iodine

The solubility of iodine and bromine in water-immiscible organic solvents is well known. Both elements are readily extracted from aqueous solution by these solvents. Thus, the partition coefficient of bromine between water and carbon disulphide and carbon tetrachloride is about 80 and 30 respectively, whilst the partition coefficient for iodine from water to carbon tetrachloride is 85. Chloroform, carbon disulphide, etc., do not extract iodine so readily from its solution in

aqueous potassium iodide, due to the presence of polyiodide ions. The best known example of the use of the extractability of the halogens in analytical chemistry is in the Andrews iodate titration, where the iodine in carbon tetrachloride is used for indicator purposes.

In the well known Liepert titration, iodate is 'stepped-up' by addition of excess iodide and mineral acid, thus liberating six equivalents of iodine.

$$5I^- + IO^-_3 + 6H^+ \longrightarrow 3I_2 + 3H_2O$$

If this iodine is extracted, returned to an aqueous phase, oxidised to iodate and taken through the amplification procedure once more, the original iodide will then have produced 36 equivalents of free iodine, etc.

Recently the separation of iodine from bromine and other materials by a solvent extraction procedure has been described in a publication by Meinke.[133] The separation utilises the selective release of the elemental halogens from their various oxidation states by various reagents. Thus, if a solution contains both bromine and iodine in the form of iodides and bromides, only the iodine is extracted by carbon tetrachloride when the aqueous stratum is made 1N with respect to nitric acid and treated with a slight excess of sodium nitrate. If the aqueous phase is subsequently treated with permanganate until a slight excess of the latter is present, bromine may also be extracted using the same solvent.

The halogens in general may be extracted as chlorides, bromides, iodides, and fluorides in association with metals such as iron[111], indium, and tin, etc., but obviously such a method can only be of value qualitatively. The 'missing' halogen astatine can also be extracted in its zero valency state in a manner similar to iodine and bromine.[148]

As mentioned previously, sulphur in Group VIb may be extracted very efficiently as free sulphuric acid by a solution of methyldioctylamine in xylene. Also in this sub-group are selenium and tellurium. Only trace amounts of selenium are extracted by diethyl ether from 6N hydrochloric acid, whereas large amounts (*c.* 34%) of tellurium pass into the organic phase. Kitahara[37] quotes 0·2% extraction of tellurium from hydrobromic acid and 0·2% from hydrofluoric acid, but gives 3·1% extraction for selenium from 40% hydrofluoric acid. No data are given for the other acids.

The solvent extraction of the alkali metal salts belongs to the field of solid-liquid extraction[27] and, therefore, is not dealt with here.

In conclusion, the author would like to draw the attention of readers to the annual reviews on extraction in the journal *Analytical Chemistry* by Craig,[150] and to the comprehensive papers by Irving,[149, 38] Furman, Mason and Pekola,[17] Kitahara,[36, 37] Morrison,[41] P. W. West[9, 40, 95, 98] and his co-workers, and also to many others mentioned repeatedly throughout these paragraphs. As noted in the opening pages, this review is far from complete, but it may serve to show that, at present, solvent extraction methods of separation are available for the removal from aqueous solution of practically all the elements commonly met with in inorganic analysis.

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A New Etching Reagent for Steel and Cast Iron

(*Continued from page* 96)

completely after such treatment. This phenomenon emphasises the suitability of the sodium bisulphite reagent for revealing chemical segregation and, possibly, lattice distortion at the grain boundaries[4]. The same experimental procedure was repeated on stabilised low carbon steel, with similar results, as shown quite clearly in Figs. 11 and 13.

A very satisfactory etch was obtained on samples of a grey cast iron used for ingot moulds and bottom plates. Since the bisulphite does not attack cementite, it can reveal the finest line of this constituent (Fig. 14). Furthermore, areas rich in phosphorus etch a different colour, represented in the photomicrograph as a darker shade of grey. These properties make the new reagent superior to nital or Stead's reagent, as it combines the properties of the two

It should be noted that under the microscope some of the structures were colourful, and every detail could be observed much more easily. A more genuine reproduction of this would be given by colour photomicrography, but this is, however, still too expensive for the average metallographer.

These few examples demonstrate the real value of this new etching reagent, which can be used in many different fields of metallographic investigation.

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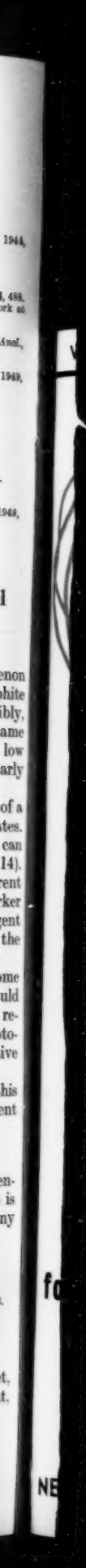
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